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SUBMICRON DISPERSION OF UO_2 IN TUNGSTEN [U]

R. V. BOWERSOCK and G. A. LAST

Sponsored by National Aeronautics and Space Administration

AUGUST, 1965

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ABSTRACT

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Under the sponsorship of the Lewis Research Center, an experimental program was conducted to determine the feasibility of producing a submicron dispersion of UO_2 in tungsten by coprecipitation. Feasibility was established; however, difficulties were encountered in maintaining the submicron particle size since subsequent thermal treatments caused coalescence of the UO_2 particles to a larger (2 to 5 μ) size. Although the process developed appears capable of producing any desired fuel loading, only powders having 10, 20, and 30 vol% UO_2 were evaluated. Minimal work was done in the fabrication of powder into usable metallic products.

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INTRODUCTION

A nuclear rocket engine for the propulsion of vehicles in outer space is being investigated by National Aeronautics and Space Administration at the Lewis Research Center, Cleveland, Ohio. The proposed engine obtains its thrust by the discharge of hydrogen gas that has been heated to greater than 2200 C by energy released in the fissioning of uranium. To meet the high temperature requirements imposed upon the fuel material, a refractory metal base fuel must be developed. This report relates to one phase of the development of such a nuclear fuel.

Under the sponsorship of the Lewis Research Center an experimental program was conducted to determine the feasibility of producing a submicron dispersion of UO_2 in tungsten by coprecipitation. Such a fuel material may have several advantages over the conventional powder blended material now being developed. These possible advantages include:

- A homogeneous product may be readily obtainable
- Because of the initial submicron size of the UO_2, subsequent working of the material into fuel plates may not result in adverse UO_2 stringering
- Fuel losses from unclad surfaces may be less because a much shallower layer of UO_2 is exposed
- Physical properties of the fuel material may be enhanced by dispersion strengthening
- The fuel material could have a considerable cost advantage over blends utilizing micronized, spherodized, or coated UO_2 particles.

The program proposed had the following objectives:

- Determine the optimum conditions for the coprecipitation of uranium and tungsten from solution.
- Determine optimum conditions required to convert the coprecipitated product into a metallic powder.

- Fabricate test specimens approaching theoretical density containing 10, 20, and 30 vol% UO_2 for metallographic examination.
- Prepare 2 to 3 kg of 20 vol% UO_2–W powder for use and evaluation by the Lewis Research Center.
- From powder containing 20 vol% UO_2, fabricate unclad test specimens 1 by 6 by 0.028 in. for evaluation by the Lewis Research Center.

Each of these objectives are discussed in this report along with the results attained during the period of this program.

SUMMARY

The feasibility of producing a submicron dispersion of UO_2 in a tungsten matrix by coprecipitation was established. This submicron dispersion was observed in metallographic examination of composites that were pneumatically impacted at 1200 C. However, difficulties were encountered in maintaining the submicron particle size since subsequent thermal treatments caused coalescence of the UO_2 particles to a larger (2 to 5 μ) size. The process developed consists of forming a flocculate precipitate of tungstic acid by treating either a solution or a slurry of ammonium paratungstate with concentrated HNO_3; an appropriate amount of uranyl nitrate is added; and the pH of the solution is adjusted to precipitate uranyl tungstate. The precipitate is dried, calcined, and reduced in hydrogen to produce a powder metal product. The process developed appears capable of producing any desired fuel loading; however, powders having only 10, 20, and 30 vol% UO_2 were evaluated in this program. Nearly 3 kg of powder containing 20 vol% UO_2 were produced for evaluation at the Lewis Research Center. A relatively coarse particle size (greater than 1.0 μ) was obtained in this product as a result of the thermal histroy during the reduction step.

Only minimal work was done in fabrication of the powder into usable metallic products. Results of the work performed were encouraging, particularly in extrusion. However, the fabrication processes studied resulted in growth of the submicron UO_2 particles.

Continued study of the coprecipitation process is recommended with particular emphasis on:

- Improvement in thermal stability of the powder by minimizing product sensitivity to agglomeration
- Evaluation of small additions of ThO_2, CaO, or Y_2O_3 for stabilizing the UO_2 and minimizing loss during thermal cycling
- Investigation of fabrication methods for producing high quality fuel plates or grids from the powder product.

OBJECTIVE NO. 1: DEVELOPMENT OF THE COPRECIPITATION PROCESS

It is reported[1] that uranyl tungstate, UO_2WO_4, is formed as a pale yellow precipitate when a solution of an uranyl salt is treated with a soluble tungstate. Menahem Merlub-Sobel[2] has patented a process for reacting aqueous solutions of appropriate salts to form insoluble precipitates to form "a nuclear fuel composition consisting of uranium dioxide in a molecular mix with the metal" (either molybdenum or tungsten). The insoluble compounds, per se, are too rich in uranium to be used directly in powder preparation; e.g., uranyl tungstate has nearly 1.5 g UO_2 per gram of tungsten which is equivalent to about 72 vol% UO_2. To obtain the desired fuel compositions of 10, 20, and 30 vol% UO_2, it is necessary to provide additional tungsten. Providing this additional tungsten simultaneously by coprecipitation, rather than by mechanical blending techniques, was the desired approach in this study.

It is worth while to compare briefly the chemical properties of uranium and tungsten.

- Tungsten is resistant to acids, but uranium dissolved easily in acids with the evolution of hydrogen.

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(1) K. C. Li and C. Y. Wang. Tungsten, Reinbold Publishing Corporation, 1955. 3rd ed., p. 286.

(2) U. S. Patent 3,102,848, September 3, 1963. Nuclear Fuel Compositions and Methods of Making the Same, Menahem Merlub-Sobel, Jersey City, New Jersey, assignor to Curtiss-Wright Corporation, A Corporation of Delaware.

- WO_3 is soluble in alkali and insoluble in acid, but the reverse is true for UO_2.
- WO_3 can be reduced to the metal by hydrogen; UO_2 is relatively stable in hydrogen.

Whereas the first two departures in chemical behavior tend to complicate the coprecipitation process, it is the difference in behavior in a hydrogen atmosphere that makes the overall concept possible.

As a basis for the selection of starting materials and chemical reagents, consideration was given only to those chemicals that produced nonmetallic salt that would be decomposed into gaseous byproducts either by calcination or during hydrogen reduction. Therefore, ammonium tungstate and uranyl nitrate were logical choices for starting materials and HNO_3 and NH_4OH for pH control. In the initial work ammonium paratungstage, APT, was used. Figure 1 illustrates the effect of pH on the room temperature solubility of APT (see Appendix for lot analysis). Since uranium is insoluble in basic solutions and since increasing the pH decreases the solubility of tungsten, the initial coprecipitation experiments involved starting with a saturated solution of APT (pH 6); adding uranyl nitrate, UNH, solution; then adjusting the solution to pH 9 with NH_4OH. Although a product of desired composition could be obtained in this manner, the uranyl tungstate formed initially was flocculent and less dense than the excess tungsten precipitated by raising the pH. As a result, stratification occurred during separation of the precipitates from the supernate. However, the feasibility of producing a submicron dispersion of UO_2 was demonstrated in these early experiments. Figure 2 is a photomicrograph of a compact made from 17 vol% UO_2–W powder produced by this process. The precipitates were reduced in hydrogen at 900 C and compacted by pneumatic impaction at 1200 C. Although the majority of the UO_2 particles were less than 0.5 μ diam, the macroscopic dispersion of the UO_2 was not uniform. Modification of the process was needed to prevent stratification due to the difference in density of the two precipitates.

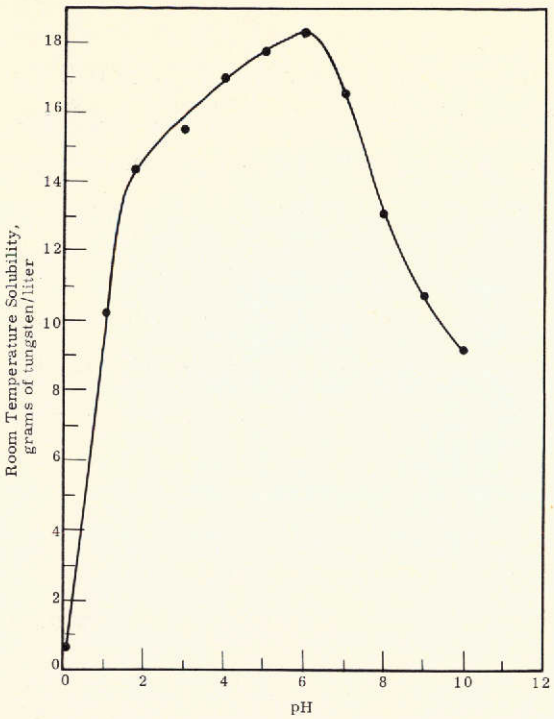

FIGURE 1

Room Temperature Solubility of Ammonium Paratungstate

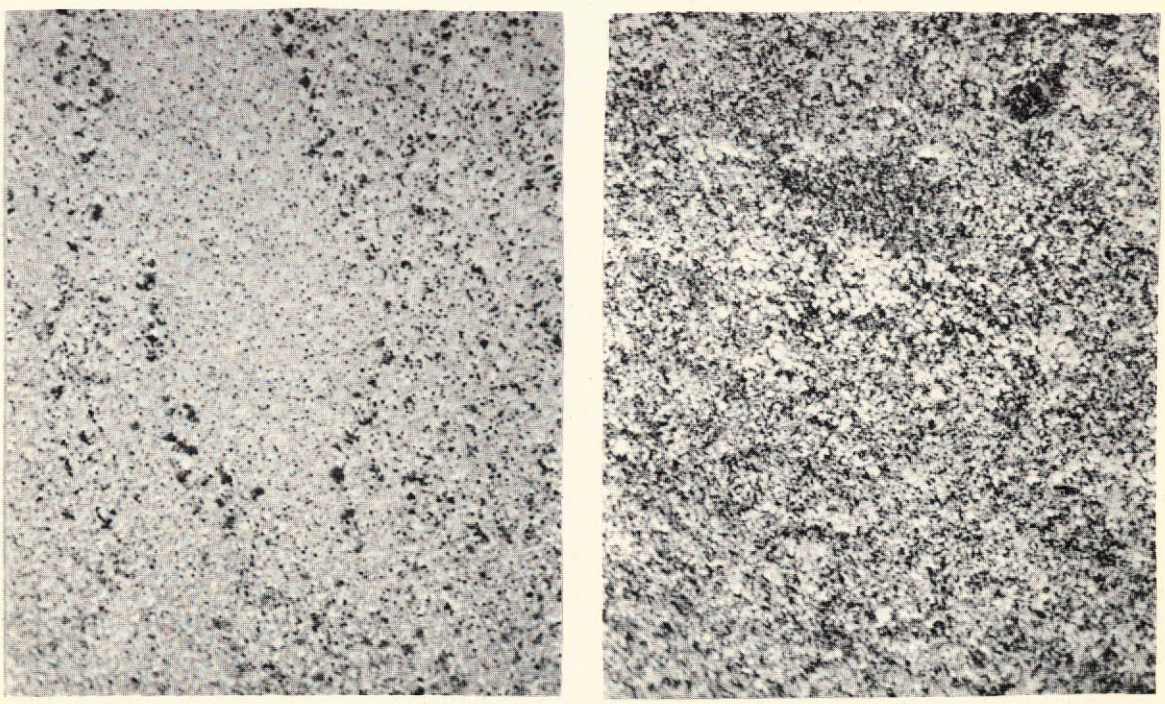

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FIGURE 2

17 vol% UO_2–W As-Compacted at 1200 C
Etchant: Murakami's Etch [10 g KOH; 10g K_3 $Fe(CN)_6$; 100 ml H_2O]

Two small specimens of the as-compacted material (Figure 2) were heated in vacuum to determine what effect elevated temperature would have upon the dispersed phase. One specimen was heated to 1500 C for 4 hr and the other heated to 2000 C for 20 min. In both, grain growth occurred to the extent that the dispersion no longer was submicron. The effect elevated temperature has on the typical grain size is summarized in Table I. Photomicrographs of the annealed material are shown in Figure 3. Thermal instability of the product will be discussed further in other sections of this report.

TABLE I

EFFECT OF TEMPERATURE UPON TYPICAL GRAIN SIZE

	UO_2 Phase, μ	Tungsten, μ
As-Compacted at 1200 C	0.5	1 to 2
4 hr Anneal at 1500 C	2 to 3	3 to 4
20 min Anneal at 2000 C	5 to 7	7 to 9

Additional solubility studies in which both uranium and tungsten were present showed that both elements could be precipitated in acid solutions as shown in Figures 4, 5, and 6. No claim is made that these are true equilibrium curves; in fact, later work showed that in some cases the precipitation process is quite temperature sensitive and is affected by techniques employed during the coprecipitation process. The curves do, however, provide a good guide in establishing the optimum pH value to use. Optimum pH is defined as that value where both uranium and tungsten losses in the supernate are minimized. For fuel material containing at least 20 vol% UO_2, the optimum pH range is between 3 and 4; and for fuel loading containing 10 vol% UO_2, the optimum range is pH 2 to 3. It is of interest to note that the solubility of tungsten is not reversible after the flocculent H_2WO_4 has formed. The dotted line on the left of Figure 4 shows the solubility of APT as pH is reduced (see also Figure 1).

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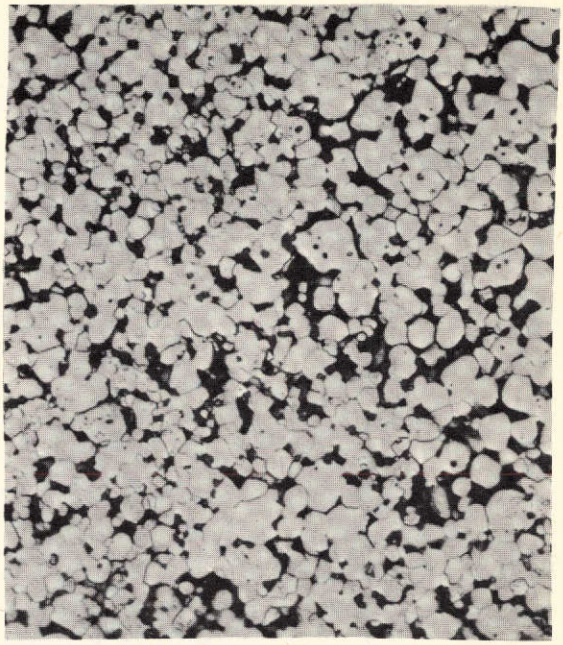

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A. Vacuum Anneal at 1500 C for 4 hr

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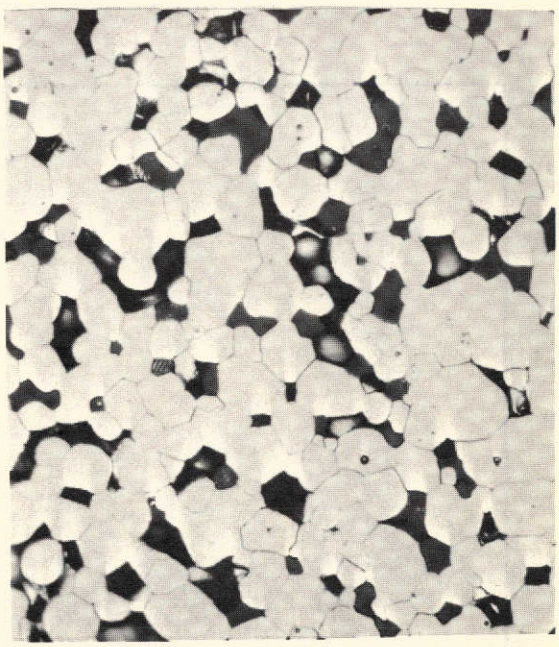

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B. Vacuum Anneal at 2000 C for 20 min

FIGURE 3

Effect of Elevated Temperature on 17 vol% UO_2–W Compact

Neg. 4L-444B; 4L-444C; 4L-443A; 4L-443B

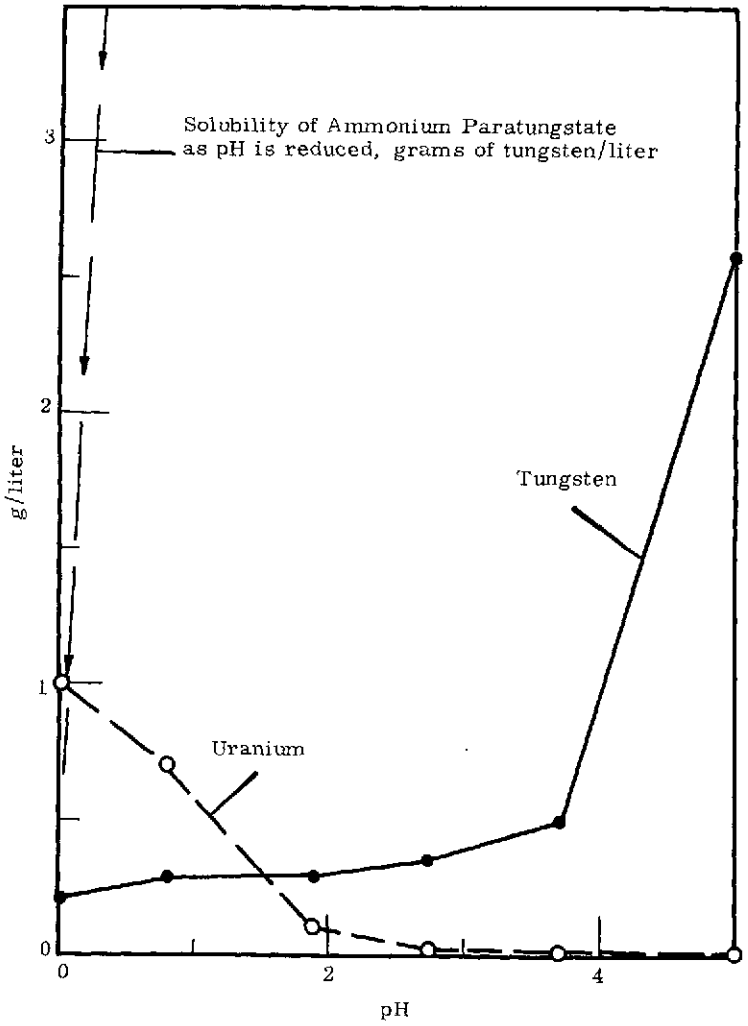

FIGURE 4

10 vol% UO_2–W Solubility Curve

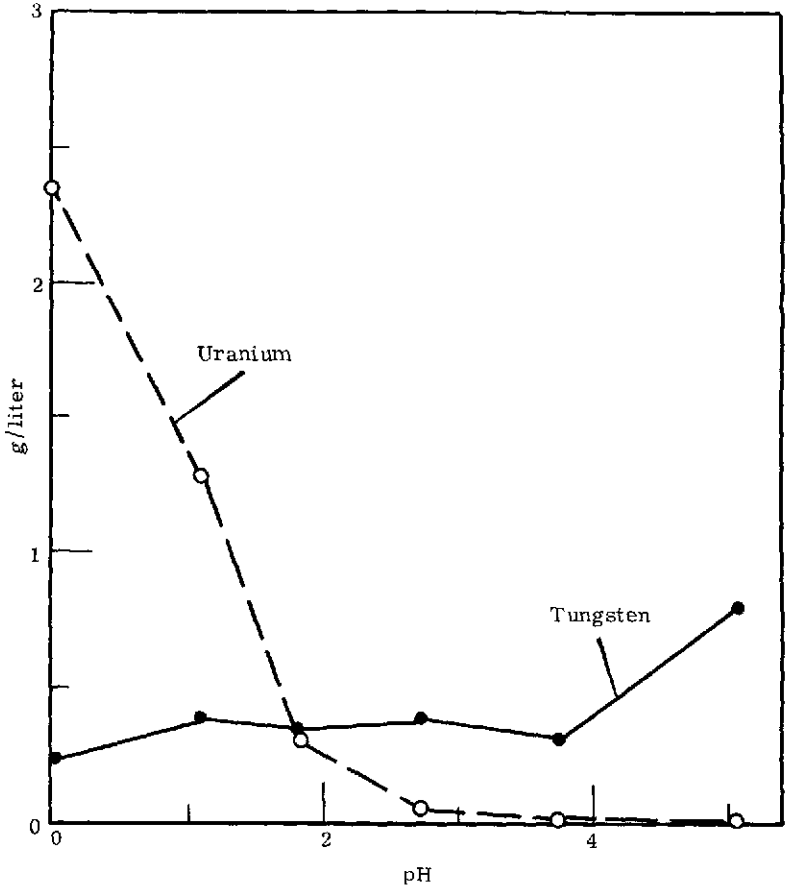

FIGURE 5

20 vol% UO_2–W Solubility Curve

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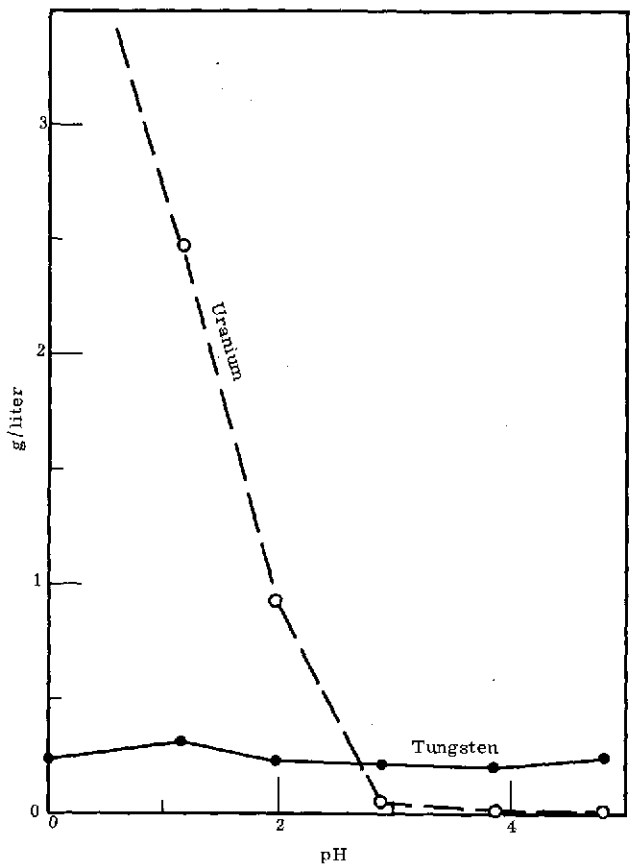


FIGURE 6
30 vol% UO_2–W Solubility Curve

From the above solubility data a revised coprecipitation process was developed and is diagrammed in Figure 7. The revised process is described below.

Step 1: A saturated solution of ammonium paratungstate is treated with HNO_3 to flocculate the tungsten as orthotungstic acid. This is achieved by reducing the pH to zero.

$$(NH_4)_{10}W_{12}O_{41} \cdot 5H_2O + 10HNO_3 + 2H_2O \rightarrow 12H_2WO_4\downarrow + 10NH_4NO_3$$

Step 2: The resulting floc is allowed to settle and the clear supernate is decanted off to reduce the liquid volume. Reduction of volume at this step where the solubility of the tungsten is at a minimum reduced the total amount of tungsten that is redissolved in later steps of the process.

Step 3: An appropriate quantity of uranyl nitrate solution is added to the highly acidic floc to provide the desired uranium to tungsten ratio. Vigorous agitation assures thorough mixing of the uranyl nitrate, which remains in solution at a pH of 0.

Step 4: NH_4OH is added slowly to adjust the pH to between 3 and 4 (for 20% UO_2 loading) which permits the precipitation of the uranium as uranyl tungstate.

$$UO_2(NO_3)_2 + H_2WO_4 + 2NH_4OH \rightarrow UO_2WO_4 \downarrow + 2NH_4NO_3 + 2H_2O$$

Step 5: The flocculent mixture of orthotungstic acid and uranyl tungstate is separated from the superate by filtering or by centrifuging.

Step 6: The resulting cake is dried and calcined to the mixed oxides.

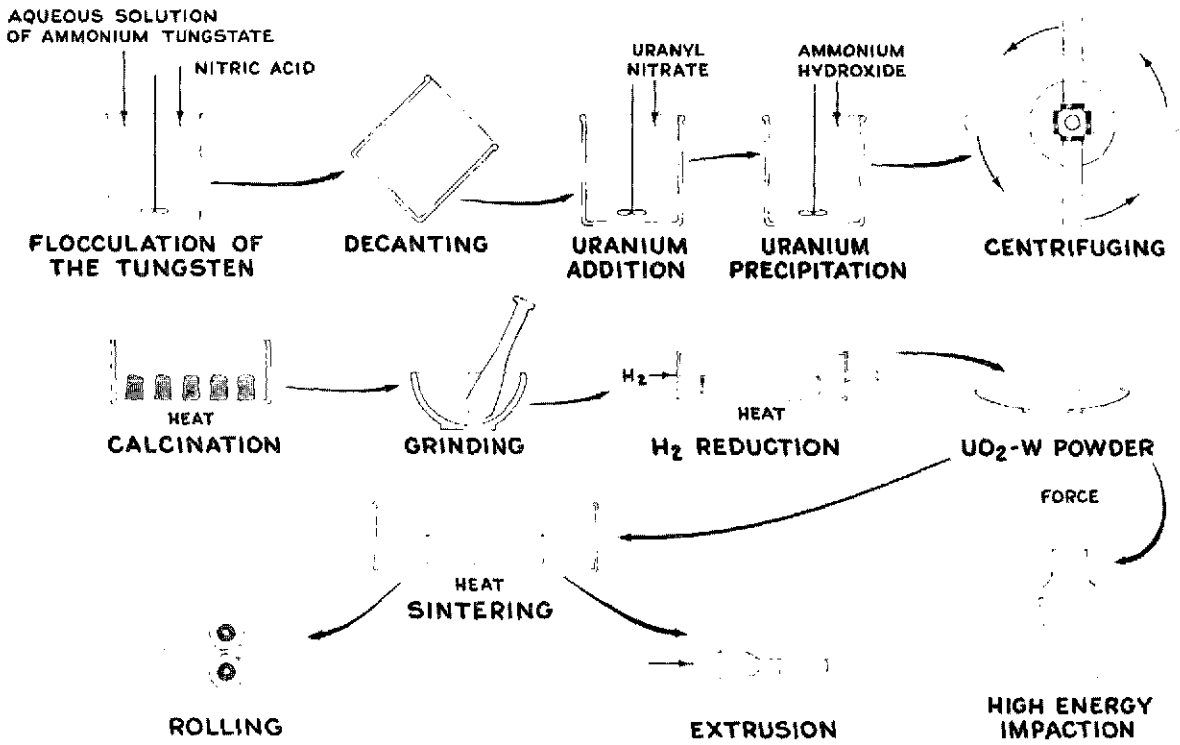


FIGURE 7

Flowsheet for the Production
of Submicron UO_2 Dispersion in Tungsten

Neg. 0641096

The key to the above process is the flocculation of the tungsten before the addition of the uranyl nitrate. The nature of this precipitate closely approaches the nature of the flocculent uranyl tungstate, and no segregation of the two appears to occur during separation of the precipitates from the supernate.

The relatively low solubility of ammonium paratungstate necessitates the handling of relatively large volumes of materials. As an alternate source of tungsten, the more soluble ammonium metatungstate, $(NH_4)_6H_2W_{12}O_{40}\cdot XH_2O$ was investigated. Although an increase in solubility of about sixtyfold can be achieved at pH 6, the solubility in the high acid range, pH 0, is approximately 400 g W/liter which is more than 500 times greater than that of the paratungstate. Increased solubility in the high acid range is undesirable during the flocculation step because of the relatively large quantity of tungsten discarded in the waste solution. Also it was found that the supernate tended to be unstable and sometimes a third phase, in the form of white needle-like crystals, settled out on top of the UO_2-W coprecipitate. Because of this latter phenomenon further investigation was made using the metatungstate.

Another approach to alleviate the problems associated with the relatively low solubility of paratungstates was the use of a slurry rather than a solution of APT as the starting material in Step 1. It was found that when a slurry of APT is treated with concentrate HNO_3, flocculent orthotungstic acid precipitated thereby permitting more APT to dissolve. This process continues until all the APT has reacted with the HNO_3. The acceptibility of this modification has been confirmed by metallography of compacts made from material prepared by this technique.

In addition to permitting larger batches to be processed with existing equipment, the slurrly technique eliminated the laborious process of dissolving and clarifying of the APT solution, eliminated the reliance upon chemical analysis of the clarified stock solution to determine tungsten content of the starting material, and eliminated the settling and decanting step

(Step 2). As a result, the overall chemical processing time was cut by nearly one half.

As an integral part of the development of the precipitation process, sample analyses played a key role. W. G. Jolley of the Hanford Analytical Laboratory developed a procedure for the anlysis of uranium and tungsten in the same solution of X-ray fluorescence. This procedure saved many hours of analytical work. A brief description of the procedure has been included in the Appendix.

OBJECTIVE NO. 2: CONVERSION OF THE COPRECIPITATE TO METALLIC POWDER

Several operations are required to convert the uranium-tungsten coprecipitate into a useful metallic powder. These operations include drying, desalting, calcining, grinding, reducing, and blending. The flocculent nature of the precipitates results in considerable quantities of liquid containing dissolved ammonium nitrates to be retained in the filter cake. The ammonium nitrate remains behind as the cake dries and is removed in the desalting operation by raising the temperature above its dissociation temperature of 210 C. Further calcining at 750 C is employed to assure that only a mixture of the oxides remains. The calcined cake is ground into a fine powder to assure uniformity in the feed to the hydrogen reduction step. After reduction, several batches are blended together to provide a sizeable lot having uniform properties and composition.

It was during the reduction step that several phenomena occurred that required considerable time in evaluation of their significance. A brief explanation of the reduction process and equipment follows.

The reduction of tungstic oxide with hydrogen is a standard industrial process for producing tungsten metal powder. This process yields a high purity metal and also permits close control of particle size. During the reduction process tungstic trioxide, WO_3, passes through several states of oxidation before being reduced to the metal. The overall reaction is:

$$WO_3 + 3H_2 \rightleftarrows W + 3H_2O \quad .$$

In general, reduction begins at about 400 C and is completed at 700 to 1000 C. Particle size is the primary factor influencing the selection of the reduction temperature with larger particles produced at higher temperatures. The water content of the reducing atmosphere influences the degree of completion of the reduction process. An excess of hydrogen plus the continuous removal of the water as it forms tends to drive the reduction to completion.

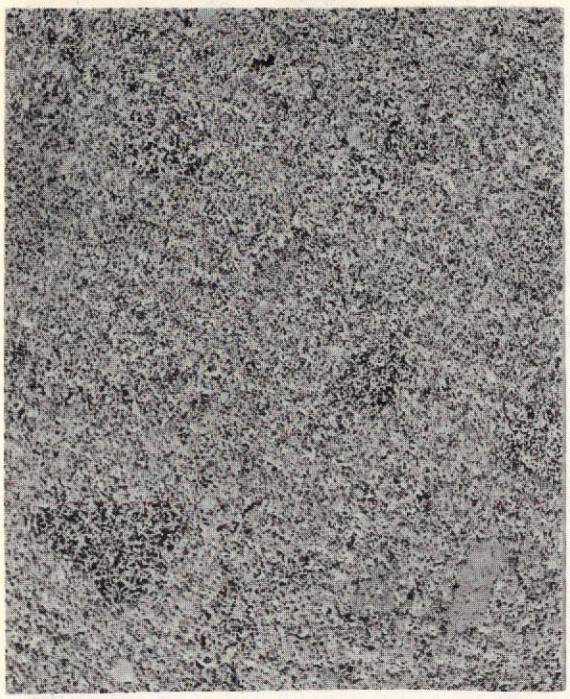
UO_2 cannot be reduced to the metal with hydrogen; however, the higher oxides, such as U_3O_8 and UO_3, are reduced to UO_2 at 650 C or above. Since the reduction requirements of both tungstic oxide and the higher oxides of uranium are approximately the same, one would assume the requirements for the mixed oxides to be similar.

The reduction equipment used in the investigation phase of the work consisted of a resistance heated laboratory tube furnace fitted with a Vycor tube. This furnace had an effective hot zone 2 in. diam by 10 in. long. A practical operating limit of 1050 C was imposed based upon the physical properties of both the Vycor tube and the nichrome heating elements.

Initial reduction were conducted using standard industrial grade tank hydrogen supplied to the furnace through several feet of Tygon tubing. A light metallic gray product was obtained from the first few runs using a reduction temperature of 900 C. However, as work progressed, the product began to exhibit a yellowish olive drab coloration and tended to be pyrophoric. Conditioning of the reduced powder under helium prior to exposure to air became necessary. In addition, a dark, smutty-appearing surface layer sometimes formed toward the gas inlet end. Increasing the reduction temperature to 1000 C and upgrading of the hydrogen supply failed to correct this apparent discoloration of the reduced product. Upgrading of the hydrogen supply by replacement of the Tygon with stainless steel tubing and by procurement of an ultra pure grade of hydrogen containing less than 10 ppm total impurities did, however, reduce the final exit dew points from -40 F to better than -60 F.

Analyses of the discolored product by both X-ray diffraction and spectrochemical techniques did not reveal any significant quantity of impurities to be present; nor did metallography of compacted specimens reveal the presence of a third phase. However, it was observed that during the early stages of reduction the exit gas gave a basic reaction to moistened pH paper. This behavior was observed even for coprecipitates that had been calcined to a constant weight at 750 C. Toward the completion of reduction, as determined by low exit dew points, the exit gas would test either neutral, or in some cases even an acidic reaction was observed. These observations suggested that nitrogen might be involved in the discoloration phenomenon. Although typical nitrogen analyses, as determined by the Micro-Kjeldahl-colorimetric method, were only on the order of 100 ppm, a special experiment was conducted aimed at reducing the nitrogen content to a minimum. The reduction was carried out in two steps. In the first step, the tungstic oxide was reduced to the dioxide at 750 C using hydrogen bubbled through room temperature water. This was done to assure that ample moisture was present to react with any residual nitrogen present after calcination. In the second step, dry, ultra pure hydrogen was used, and the reduction temperature was slowly increased to 1100 C and held for 2 hr. No significant improvement in color of the reduced product was obtained, although the nitrogen content was reduced to 34 ppm.

A final approach to the problem was to investigate the effect of still higher temperatures. The 1100 C used in the above reduction was the upper limit that could be obtained in the existing laboratory setup. Therefore, portions of this discolored powder were given further treatments in a small vacuum furnace at 1500 C for 1 hr and 1300 C for 1 hr. In both cases, a metallic gray-colored product was obtained accompanied by an increase in bulk density. Compacts were made of the discolored as-reduced material and of the two treated powders. Impaction temperature was 1050 C so as not to alter the properties of the as-reduced material. Photomicrographs of the compacted materials are shown in Figures 8, 9, and 10. The following observations can be made from this study:



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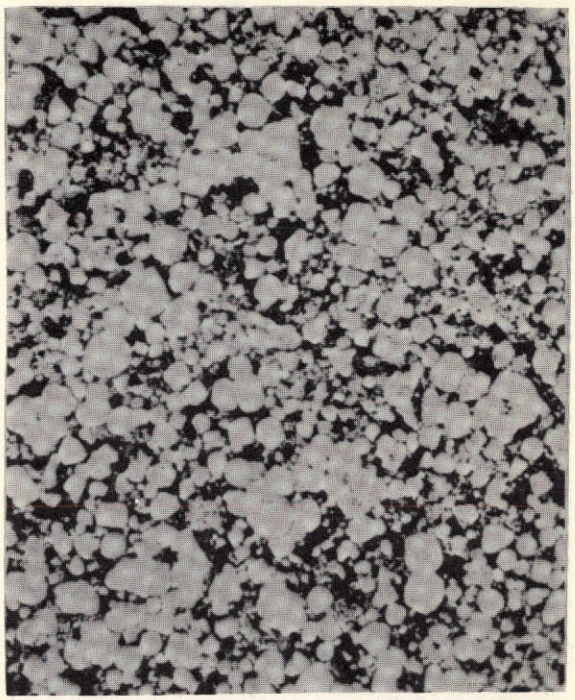

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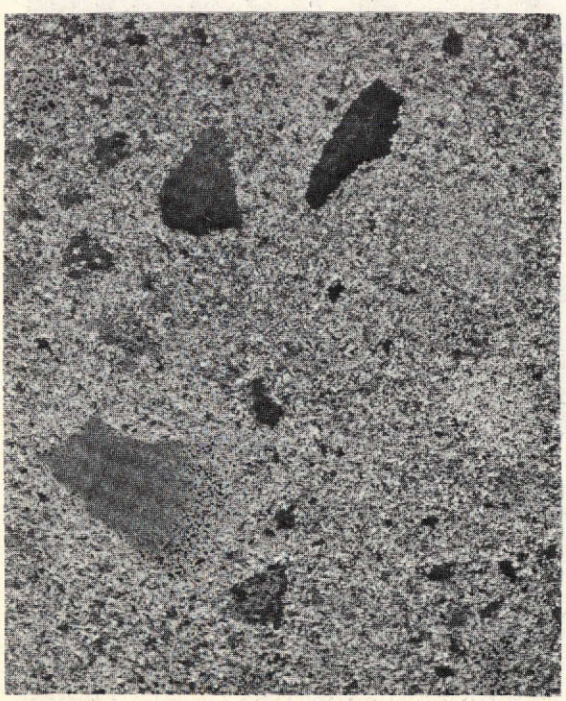

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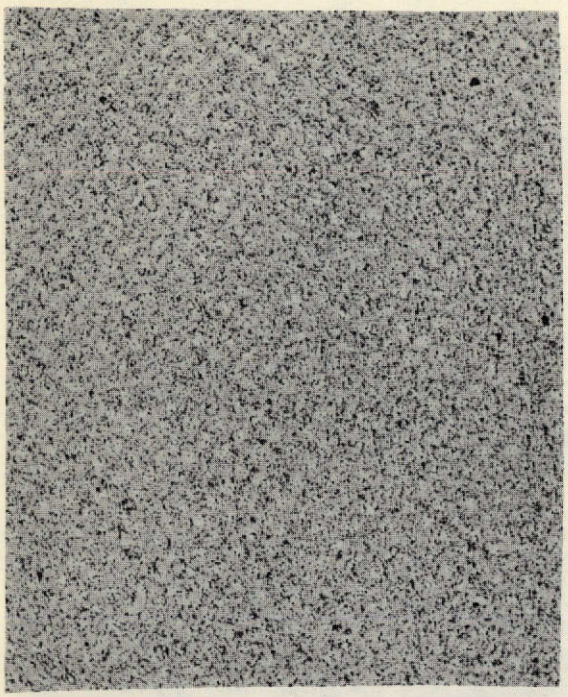
Etched (Murakami's) 1000X

FIGURE 8

20 vol% UO_2–W Compact, As-Reduced at 1100 C
4Z-4727A; 4Z-4727B; 4Z-4727C; 4Z-4727D



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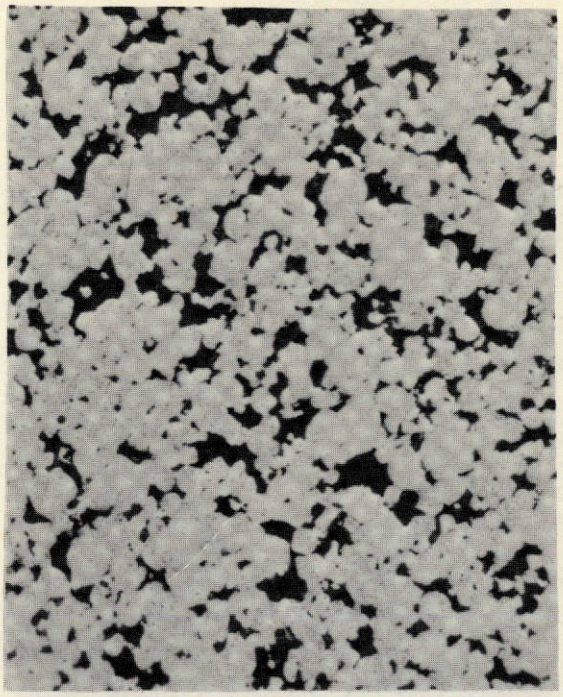

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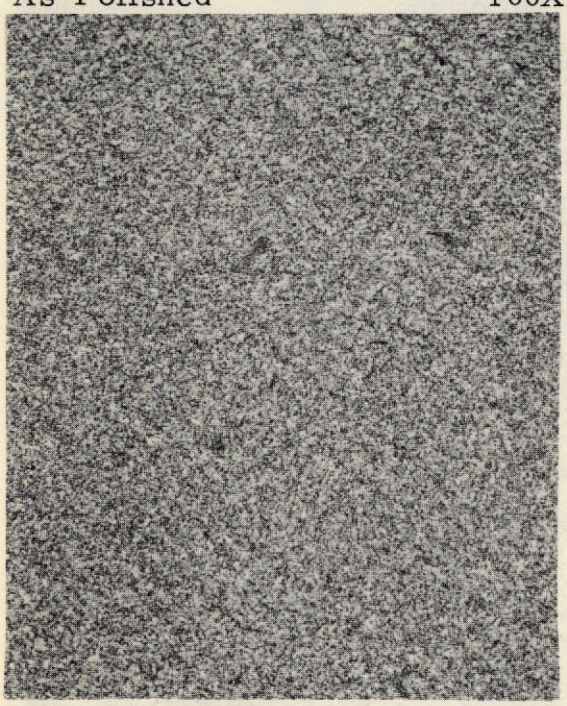

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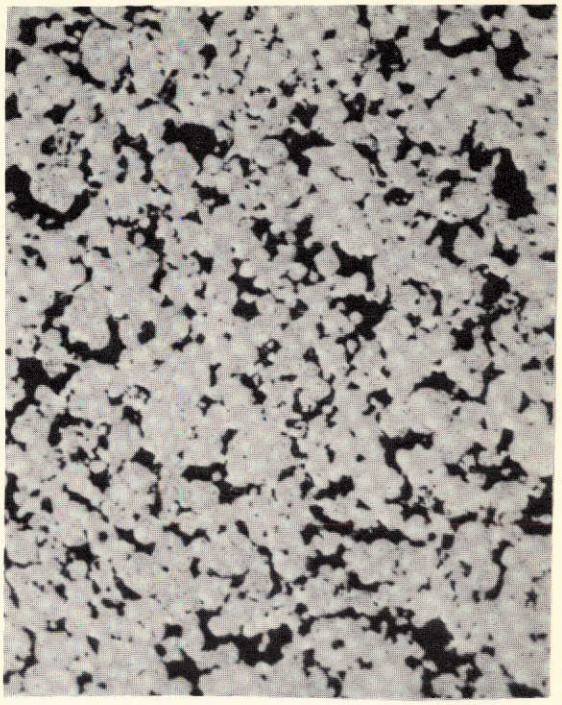
FIGURE 9

20 vol% UO_2–W Compact
(Powder Heat Treated in Vacuum at 1300 C for 1 hr Prior to Compaction)
4Z-4728A; 4Z-4728B; 4Z-4728C; 4Z-4728D

As-Polished 100X



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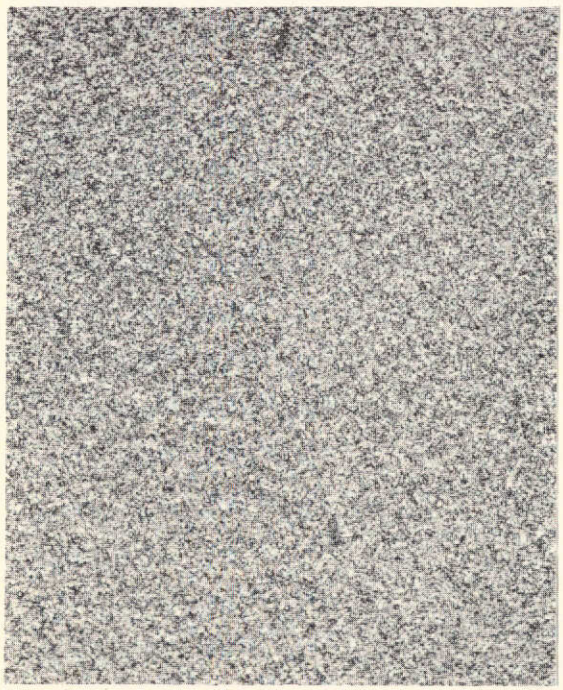

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Etched (Murakami's) 1000X

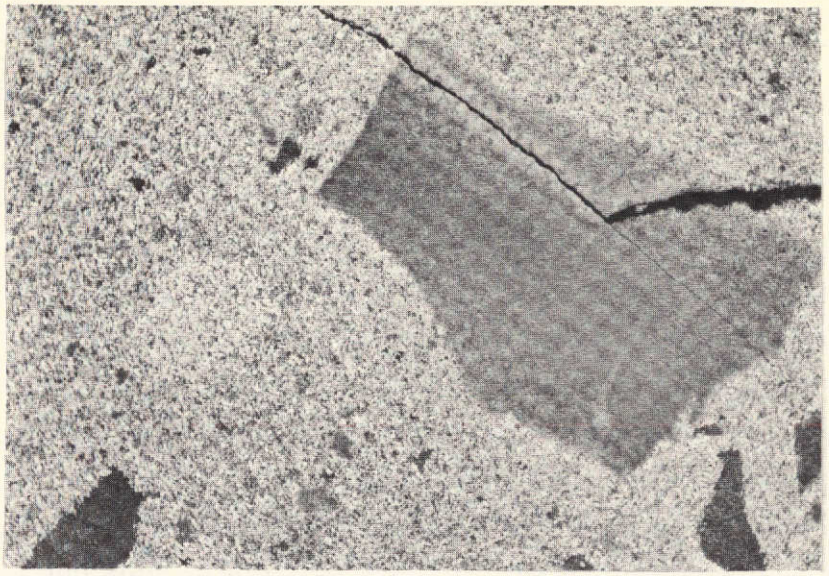
FIGURE 10

20 vol% UO_2–W Compact
(Powder Heat Treated in Vacuum at 1500 C for 1 hr Prior to Compaction)
4Z-4729A; 4Z-4729B; 4Z-4729C; 4Z-4729D

- Heterogeneity of the as-reduced compact is more evident in the as-reduced etched specimen (Figure 8). A more homogeneous structure (as viewed at 100X magnification) results from heating to 1300 and 1500 C (Figures 9 and 10).
- All three of the compacts show that the UO_2 (the dark phase) has agglomerated to where the dispersion is no longer submicron.
- Heat treating of the powder has caused the UO_2 to coalesce into a more distinct phase. The volume occupied by the coalesced phase remains essentially the same as that occupied by agglomerated particles in the "as-reduced" specimen.
- The small tungsten particles intermixed with the agglomerated UO_2 have in most cases disappeared on heating to 1300 and 1500 C.
- Heat treating has caused an increase in the average particle size of the tungsten; however, maximum size appears unchanged.
- There is little significant difference in the structure obtained with 1300 and 1500 C heat treating temperature.

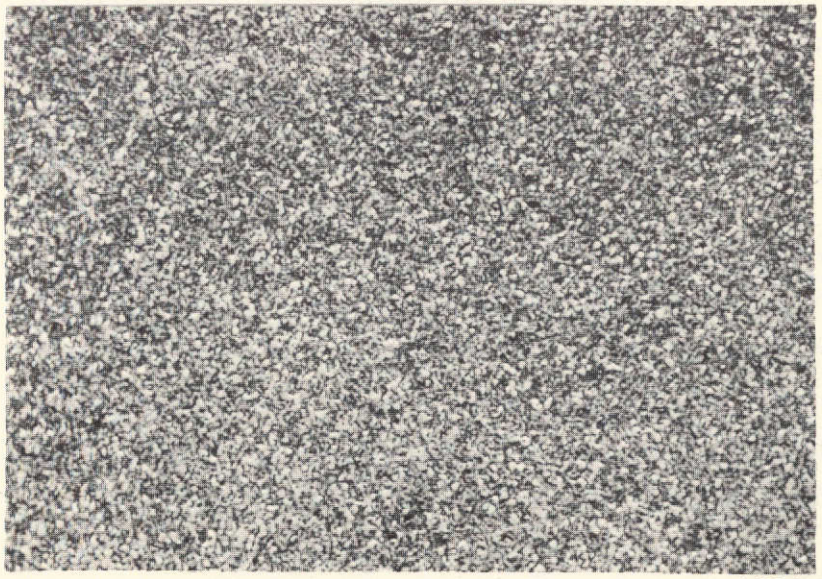
The heterogenity of the as-reduced compact is further illustrated in Figure 11. In the higher magnification photomicrograph of the large, finely dispersed area to the right of center in the upper photomicrograph, it can be seen that the desired submicron dispersion has been achieved. Why this area should differ from the bulk of the material has not been determined. However, one should keep in mind that although there are differences in texture, the macroscopic dispersion of the fuel loading is quite uniform. This is illustrated in lower magnifications of Figures 8, 9, and 10.

In summary, it is concluded that the apparent discoloration of the as-reduced product is probably not due to either impurities or incomplete reduction but appears to be due solely to particle size. It is possible that trace impurities which have segregated during the drying process may be responsible for the wide variation in particle size seen in the as-reduced powder.



Etched (Murakami's) 100X

A. 20 vol% UO_2–W Compact



1000X

B. Higher Magnification of Large, Finely Dispersed Area in Above Micrograph

FIGURE 11

Heterogenity of As-Reduced Powder
(Reduction Temperature, 1100 C; Impaction Temperature, 1050 C)
Neg. 4Z-4727G; 4Z-4727E

OBJECTIVE NO. 3: METALLOGRAPHY OF SPECIMENS CONTAINING 10, 20, AND 30 vol% UO_2

The coprecipitation process can be used to produce a fueled matrix having any desired loading of UO_2. However, one of the objectives of this program was to provide data showing how particle size and distribution were affected by varying the fuel loading in the 10 to 30 vol% range. To meet this objective it was necessary to provide densified specimens of the as-reduced powder without appreciably altering particle size, shape, or distribution. High-energy-rate impaction was selected as the best means of producing specimens because it provided higher forces yet less deformation than did more conventional processes. Previous work with W-UO_2 cermets produced by powder blending techniques had demonstrated that 98 to 99% of theoretical density could be obtained by impacting with 200,000 to 250,000 psi at 1200 C. Three batches of powder containing 10, 20, and 30 vol% UO_2 were prepared by the slurry technique. Process variables associated with the process are tabulated in Table II. The 20 vol% UO_2 powder was prepared prior to the adoption of the two-step reduction process as explained in Objective No. 2. Figures 12, 13, and 14 give the metallography of compacts made from the three powders.

The powder containing 20 vol% UO_2 was impacted first. Examination of this compact revealed that the dispersion of the fuel material was quite uniform; however, agglomeration had occurred to the extent that the dispersion no longer was submicron. Believing that the agglomeration occurred during the 1200 C preheat before impaction, the compaction temperature was reduced to 1050 C for both the 10 and 30 vol% UO_2 materials. Metallography shows that the UO_2 had agglomerated in these compacts also. One can only conclude that either agglomeration occurs at some threshold temperature below 1050 C or the agglomeration had already occurred before impaction. Further evidence of low temperature agglomeration was observed in Objective No. 4. This subject is discussed further in the Discussion of Results Section.

TABLE II

PROCESS VARIABLES ASSOCIATED WITH THE PRODUCTION OF W-UO_2 COPRECIPITATED FUEL MATERIAL CONTAINING 10, 20, AND 30 vol% UO_2

Process Variable	Nominal Fuel Loading, vol% UO_2		
	10	20	30
Batch Size, g	200	100	200
Final pH	3.47	3.75	3.50
Calcination	4 hr at 750 C	1.5 hr at 800 C	4 hr at 750 C
H_2 Reduction: Wet H_2	6.5 hr at 750 C	- -	4 hr at 750 C
	1 hr at 850 C	- -	1 hr at 850 C
	2 hr at 950 C	- -	1 hr at 950 C
Dry H_2	3.5 hr at 950 C	3 hr at 900 C 2 hr at 1000 C	3 hr at 950 C
Impaction Temperature[a]	1 hr at 1050 C	0.5 hr at 1200 C	1 hr at 1050 C
Chemical Analysis of Reduced Powder:			
wt% UO_2 (vol%)	6.39 (10.8)	(b)	19.30 (29.7)
wt% Tungsten	93.52	(b)	80.38
ppm Carbon	13	(b)	44
ppm Nitrogen	0	(b)	0

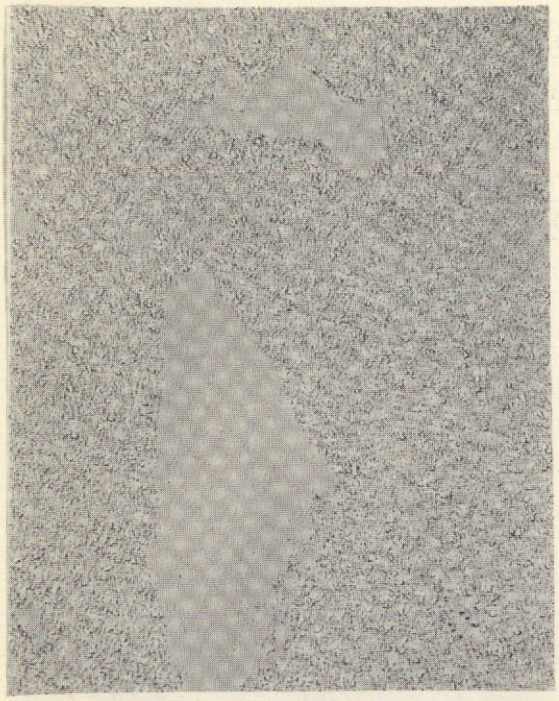
(a) Impaction Force of 250,000 to 300,000 psi common for all three materials.

(b) Powder not analyzed.

One final observation from Figures 12, 13, and 14 is that increasing the uranium content to 30 vol% has had no appreciable effect upon the overall structure of the compacts.

OBJECTIVE NO. 4: PREPARATION OF 2 TO 3 kg of 20 vol% UO_2-W POWDER

Powder containing 20 vol% UO_2 was to be prepared by the coprecipitation process for evaluation at the Lewis Research Center. Limited solubility of the ammonium paratungstate, voluminous nature of the flocculent precipitates, and slow filtering rates prevented producing a large quantity of product in a single batch with the equipment available. Therefore, to produce a sizeable lot of material, blending of several smaller batches was necessary. Initially, 20 liters of a near saturated solution of APT



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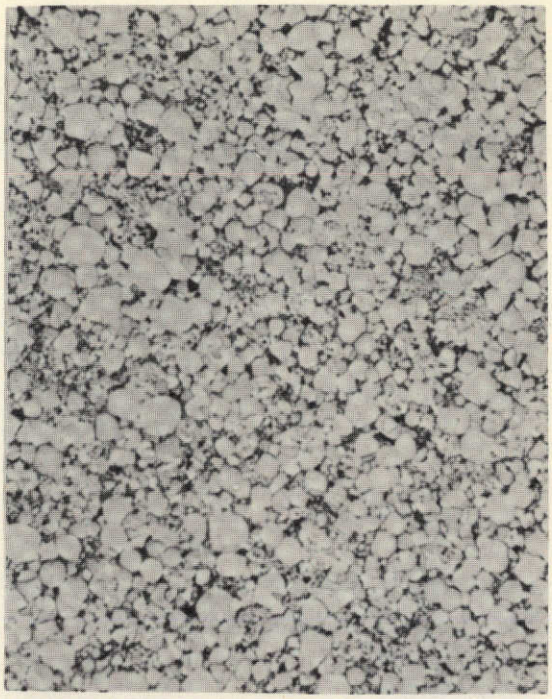

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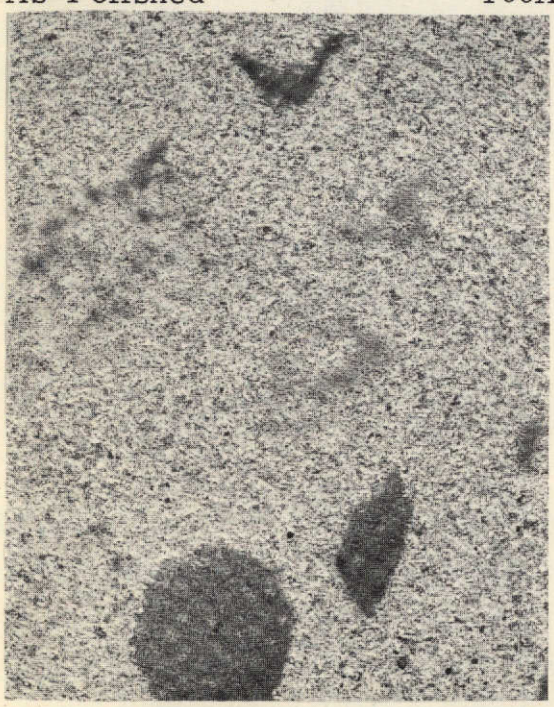

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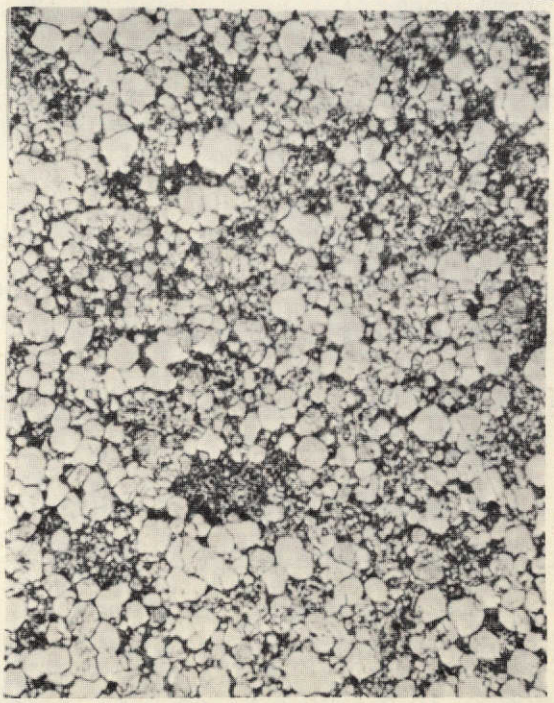

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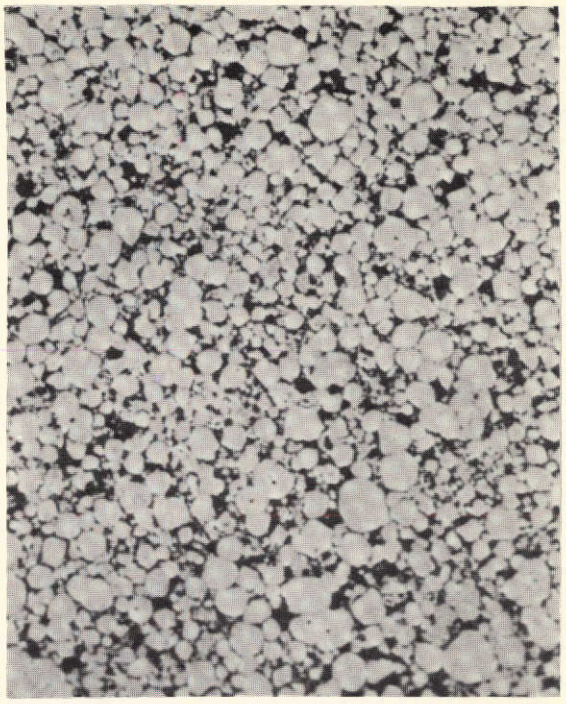
FIGURE 12

10 vol% UO_2–W Compact

Neg. 4Z-4929A; 4Z-4929B; 4Z-4929C; 4Z-4929D

As-Polished 100X



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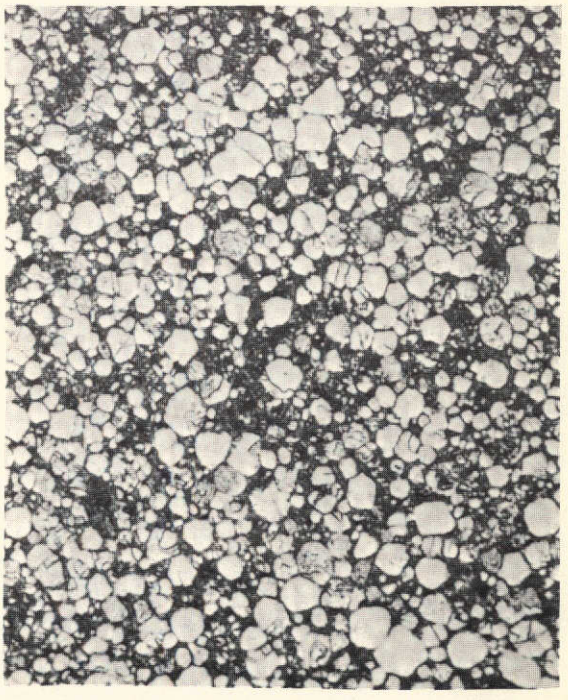
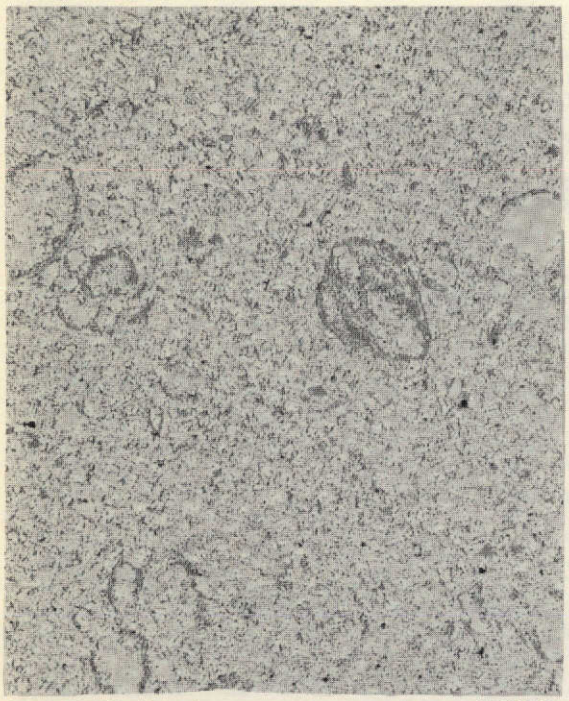


Etched (Murakami's) 1000X

FIGURE 13

20 vol% UO_2–W Compact

Neg. 4Z-5457A; 4Z-5457B; 4Z-5457C; 4Z-5457D



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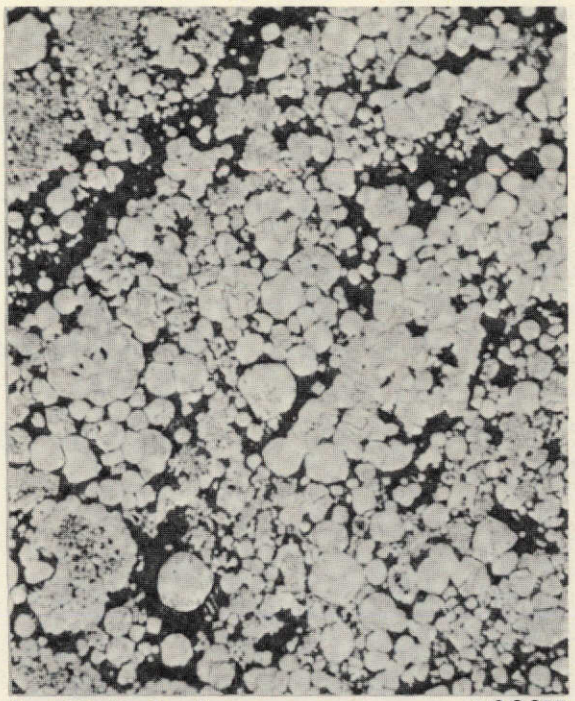

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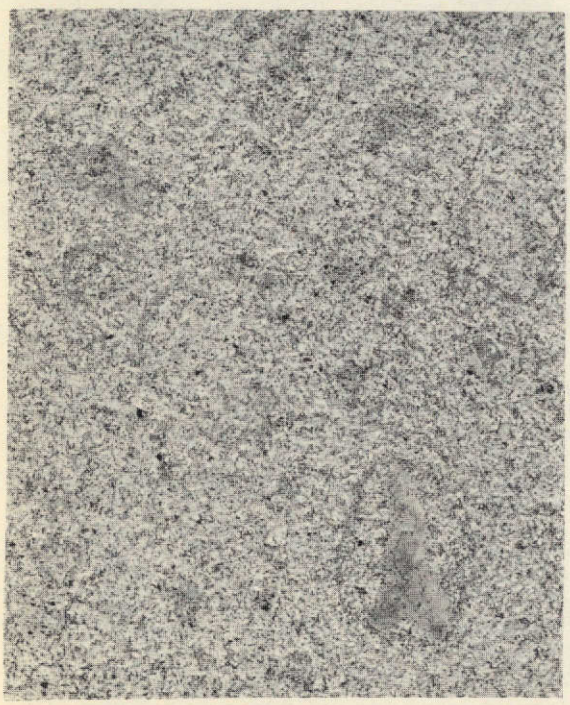

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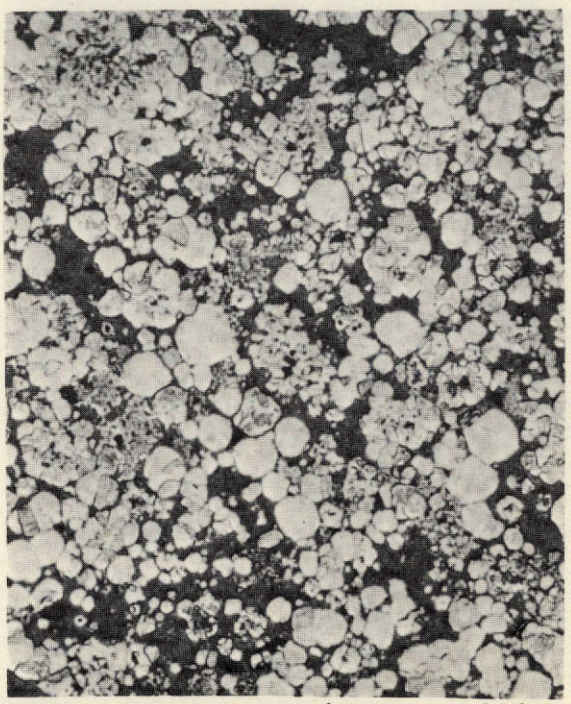

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FIGURE 14

30 vol% UO_2–W Compact

Neg. 4Z-4928A; 4Z-4928B; 4Z-4928C; 4Z-4928D

was used as the starting material producing approximately 300 g of final product. Development of the slurry technique permitted an increase in batch size to 500 g utilizing the same laboratory equipment. Approximately 4 kg of product were made by the combined processes. The coprecipitates were dried at 110 C, desalted at 250 C, and calcined for 4 hr at 750 C. The calcined material was ground and blended in a tungsten lined ball mill. The powder was difficult to grind because it tended to pack in the corners and to cake onto the wall of the mill. Bulk density of the ground powder was approximately 2.6 g/cm^3.

Data developed under Objective No. 2 indicated it would be desirable to reduce the oxides in two steps, followed by a short treatment at elevated temperature to produce a metallic colored product. The proposed reduction process was to reduce the tungsten to the dioxide at 750 C, then raise the temperature to 950 C and hold until the reduction was complete, as indicated by a low exit dew point. A final treatment at 1300 C was selected to provide a metallic colored product. In the actual reduction, difficulties developed requiring slight modification of the proposed procedure as will be discussed below.

The small glass tube furnace used during development did not have sufficient capacity to effectively handle the 4849 g of mixed oxides. Therefore a larger, higher temperature furnace was used. An initial charge of 2925 g was loaded, and the temperature was raised slowly (50 C/hr) to 750 C. This temperature was held for 6 hr, and then the temperature was raised to 950 C at a rate of 200 C/hr. After 21 hr the exit dewpoint still remained above 60 F. This was unexpected because 4 to 6 hr at temperature had been sufficient to complete reduction in the smaller furnace. The run was terminated, and the furnace was allowed to cool overnight to permit removal of the powder for inspection. The powder was found to be a pale yellow except that around the fringes, where the powder depth was less, the powder was a metallic gray. Although the powder appeared to be incompletely reduced, a weight loss of 566 g had occurred. Assuming

some loss due to removal of absorbed moisture, the observed loss compared favorably to the theoretical value of 544 g expected for complete reduction. However, since the exit dew point had still remained high further reduction seemed advisable.

The above powder was mixed with the remaining calcined material, and the mixture was returned to the furnace for reduction. Not until after the start of this second run was it discovered that the dryer on the gas inlet was not operating properly. What effect this had on the dew points of the first run cannot be assessed. A new dryer was placed in service. For the second reduction, the furnace temperature was raised directly to 950 C at a rate of 100 C/hr. After 10 hr at temperature the dew point had dropped from above 60 F to 36 F. The dew point continued to drop slowly requiring 18 more hours to reach -5 F and another 26 hr (54 hr total) to reach -27 F. Since the fringe material had been metallic gray in the first run, the reduction was terminated at this point without treating at elevated temperatures. On cooling overnight the exit dew point was -55 F when the material was removed for examination. The powder was found to be still a yellowish brown, and the inlet end ignited when it was exposed to air. The boat was returned to the furnace and flooded with helium to extinguish the fire. After purging with helium for 5 hr, the material again was removed, and again the burned area ignited. The material was returned to the furnace and the burned powder was reduced at 950 C for 15 hr. The temperature was then raised to 1300 C and held for 2 hr in an attempt to reduce the pyrophoric tendency of the powder and also to produce a metallic gray product. After cooling overnight followed by a 30 min purge with helium, the powder was removed, and once again it ignited. The powder was conditioned further with helium for 2 hr after which time the powder was removed and handled in air without ignition. The powder was an olive color, except for the fringe which was a dark slate gray. The final weight was 3896 g—giving an actual weight loss of 953 g compared to an expected theoretical loss of 903 g.

The reduced powder was ground and blended for 4 hr in a tungsten mill. During milling, the color of the powder changed to a dark, slate gray and gained approximately 50 g in weight. The ground powder was placed in a glass jar for storage. Several days later it was observed that drops of moisture had collected around the top of the jar. Using the small glass tube furnace, the powder was given a final cleanup in ultra pure hydrogen at 750 C until the exit dew point was -50 F or lower. This operation required from 2 to 3 hr at temperature, depending upon the size of batch being processed. There was no apparent change in color, although a weight loss of 50 g occurred. Portions of the material were used in evaluation of the product and in the impaction of plates (See Objective No. 5). The remaining 2925.5 g were shipped to the Lewis Research Center.

In evaluating the product, a sample of the ground oxides, a sample of the blended reduced powder, and a sample of the reduced powder after final cleanup in ultra pure hydrogen were analyzed. Carbon and nitrogen determinations are tabulated in Table III.

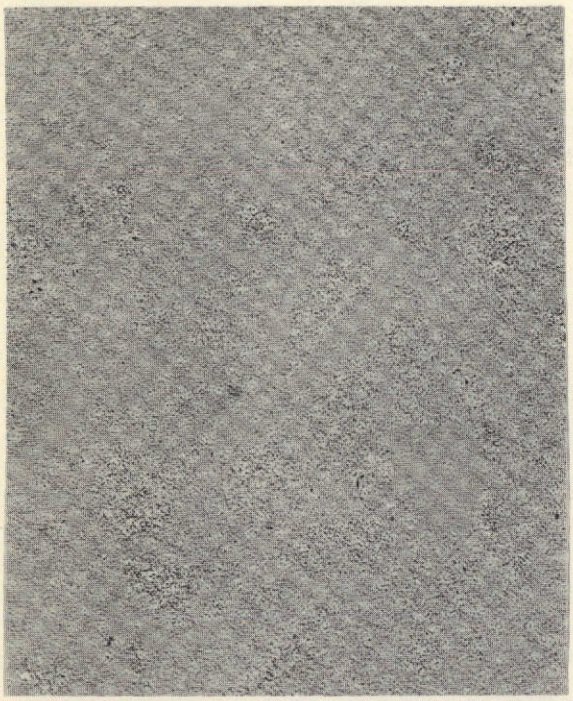
TABLE III

CARBON AND NITROGEN ANALYSES OF THE 20 vol% UO_2-W POWDER

	C, ppm	N, ppm
Mixed Oxides	2	72
After Reduction	154	55
After Final Cleanup	23	45

No explanation is given for the carbon pickup in the as-reduced sample. No significant quantity of impurities was detected by spectrochemical analyses of either the mixed oxides or the reduced product. Composition of the final product was 87.44% tungsten and 11.53% UO_2 (18.74% by volume).

A sample of the gray powder from around the fringe of the first reduction run and a sample of the final product were impacted into dense specimens for metallography. Impaction temperature was 1050 C. Metallography of these specimens is given in Figures 15 and 16. Figure 15 shows that agglomeration of the UO_2 phase has occurred; however, there still remain areas



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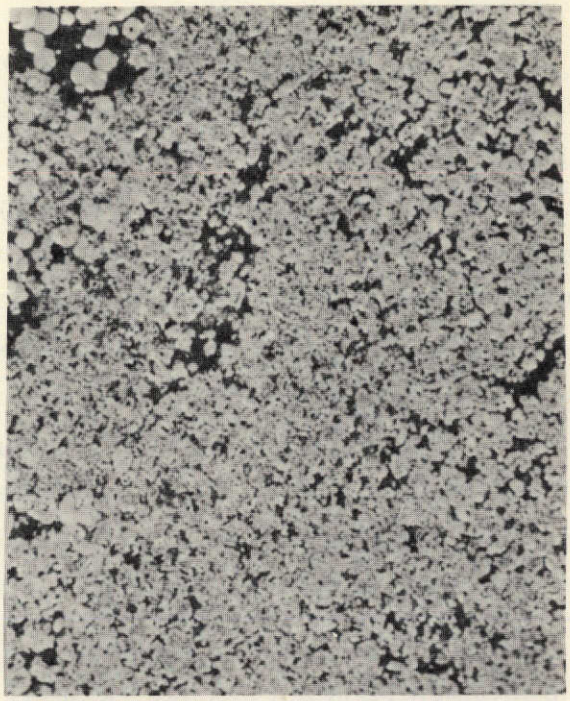

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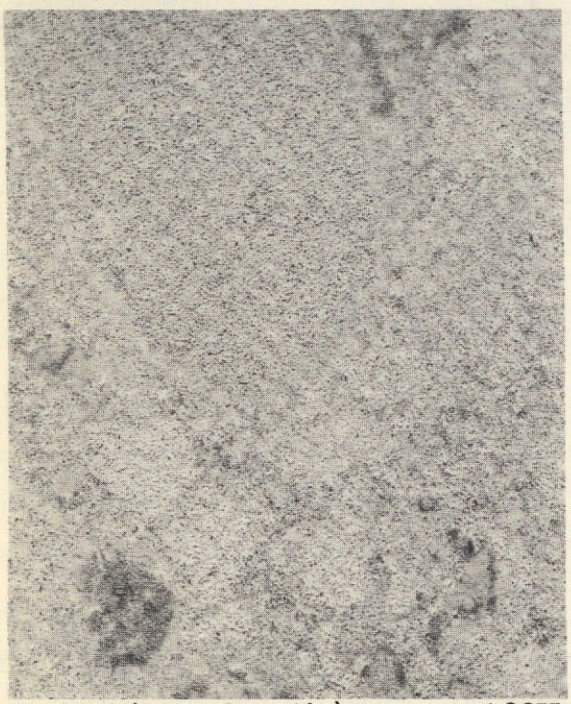

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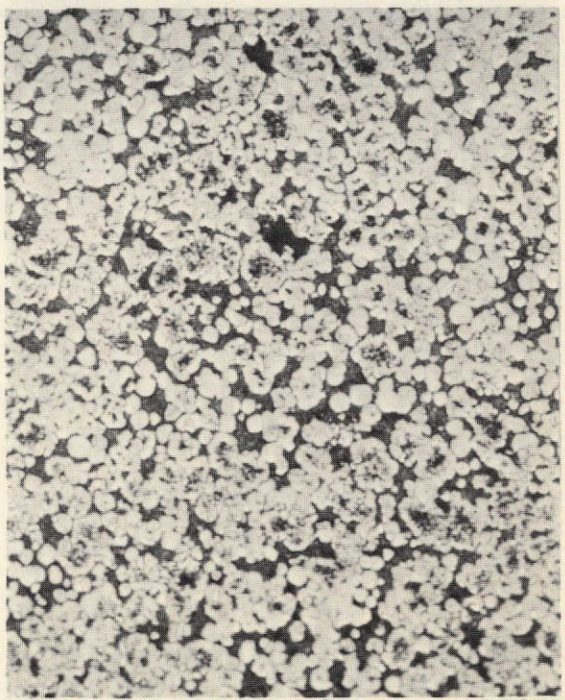
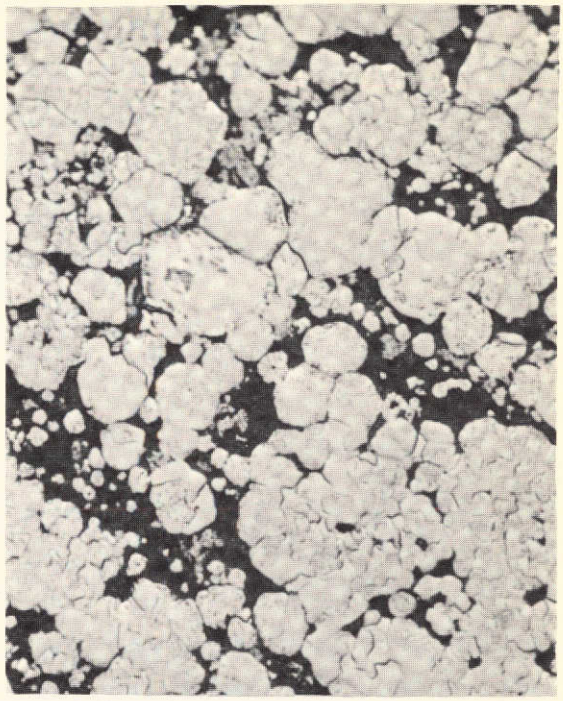


Etched (Murakami's) 1000X

FIGURE 15

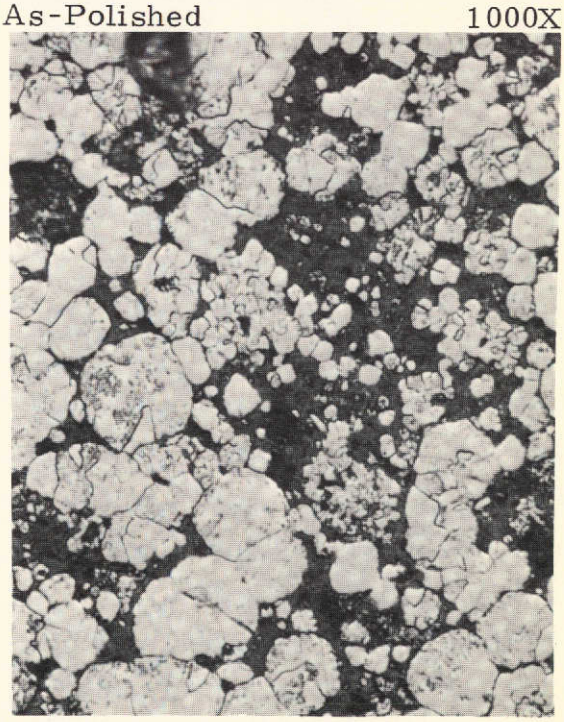
20 vol% UO_2-W
(Reduction Time: 21 hr at 950 C)
Neg. 4Z-4927A; 4Z-4927B; 4Z-4927C; 4Z-4927D

As-Polished 100X



As-Polished 1000X

Etched (Murakami's) 100X



Etched (Murakami's) 1000X

FIGURE 16

20 vol% UO_2
(Reduction Time: 90 hr at 950 C; 2 hr at 1300 C)
Neg. 4Z-4926A; 4Z-4926B; 4Z-4926C; 4Z-4926D

where the dispersion is submicron. In Figure 16, the agglomeration has progressed to the point that both the tungsten and the UO_2 particle sizes are appreciably larger than the submicron dispersion desired. Since both specimens were made from the same lot of material and both have essentially the same impaction history, the difference in agglomeration can only be attributed to the difference in reduction history. Although treating of the material at 1300 C for 2 hr undoubtedly attributed to the gross agglomeration shown in Figure 16, previous work in which powder was treated at 1500 C (See Figure 10) did not approach the degree of agglomeration seen in this specimen. Therefore, it can only be concluded that the gross agglomeration resulted from the extended time this powder was at 950 C. Although the material had agglomerated more than any other material made in this study, time limitations on this program did not permit further development work nor processing of another large quantity of material.

OBJECTIVE NO. 5: FABRICATION

The final objective of this program was to fabricate unclad test specimens, 6 by 1 by 0.028 in., containing 20 vol% UO_2. These specimens were to be shipped to the Lewis Research Center for evaluation. Because of the adverse effect of elevated temperature on UO_2 particle size, the standard method of fabricating plates by sintering "green" compacts at 1730 C was not investigated. Fabrication techniques were sought in which either the maximum temperature did not exceed 1200 C or the time at elevated temperature was relatively short. Two techniques that appeared to offer the best chance for success were pneumatic impaction and extrusion. Considerable development work in the field of pneumatic impaction already had been performed at Hanford. Also in previous work the feasibility of extruding powder compacts had been demonstrated. Both techniques were investigated and are discussed below.

Plate Fabrication by Pneumatic Impaction*

Two attempts were made to produce plates by pneumatic impaction. A portion of the 20 vol% UO_2–W powder produced under Objective No. 4 was used as the starting material. The as-reduced and blended powder was given a final clean-up in ultra pure hydrogen then loaded and sealed within a stainless steel container without exposure to air. A schematic diagram of a loaded impaction container is shown in Figure 17. Provisions were made to allow a small flow of hydrogen to permeate the powder during preheat. Total time in the 1200 C preheat furnace was 30 min after which both the hydrogen supply and the evacuation tubes were pinched off, and the container was impacted at 250,000 to 300,000 psi. The first container was loaded to produce a plate 0.028 in. thick by approximately 3 3/4 in. diam. Although this would not produce the desired 6 in. length it was the largest container the existing tooling could accommodate. Attempts to remove the plate from the impaction container were unsuccessful due to adherence of the cermet to the polished stainless steel shims. The second container was loaded to produce a 0.080 in. thick plate. Again it was difficult to separate the cermet from the stainless steel; however, mechanical separation was achieved as shown in Figure 18. Plate thickness varied from about 60 to 90 mils.

One of the small fragments was annealed in vacuum at 2500 C for 1 hr. The effects of this anneal are shown in Figures 19 and 20. The agglomerated UO_2 phase of the as-compacted plate has consolidated into single particles with considerable rounding of their edges. The tungsten phase, however, shows no significant change. A loss in UO_2 from the surface has occurred to a depth of about 12 mils. Higher magnification shows that the outer band is completely free of uranium and contains voids where the UO_2 had been. Inside of this band the UO_2 is unaltered.

* Performed by W. J. Lackey, Jr., Ceramics Research and Development, Battelle-Northwest.

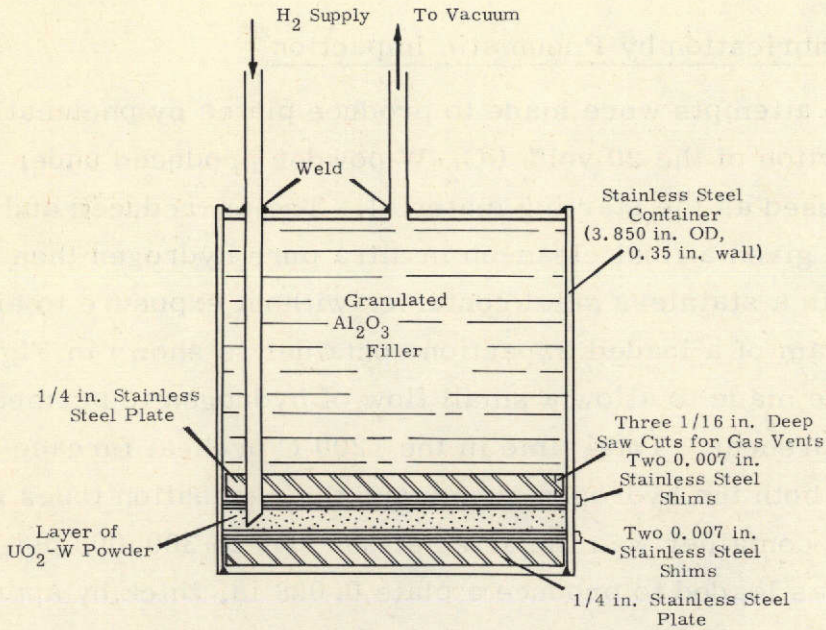


FIGURE 17

Schematic of Loaded Impaction Container

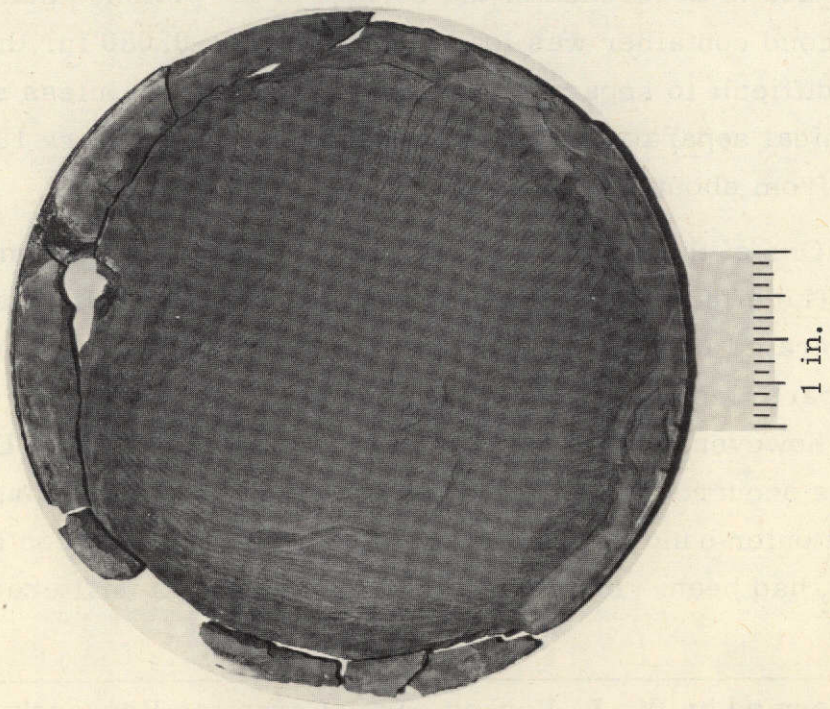
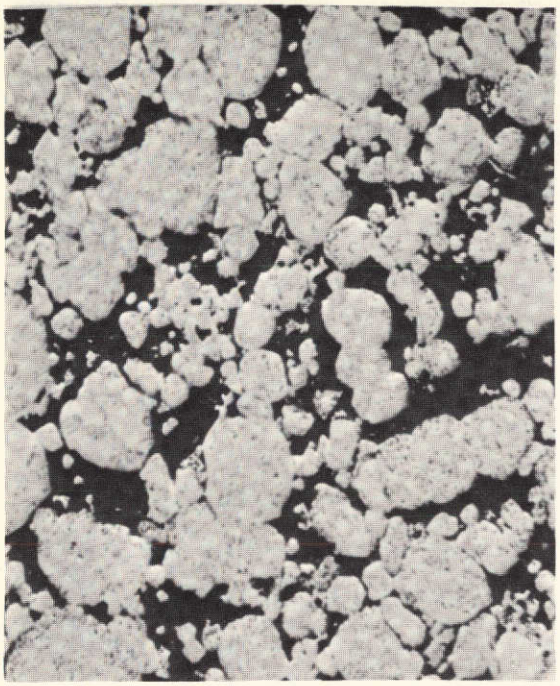


FIGURE 18

80 mil Plate As-Impacted from 20 vol% UO_2–W Powder

Neg. 5Z-5056



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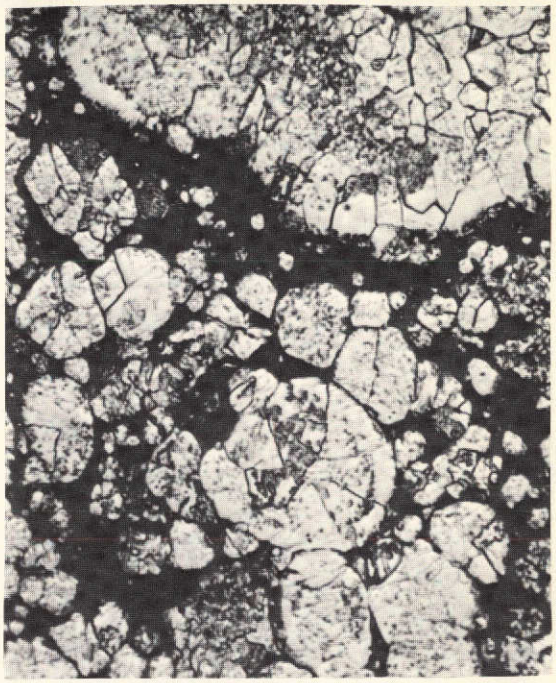

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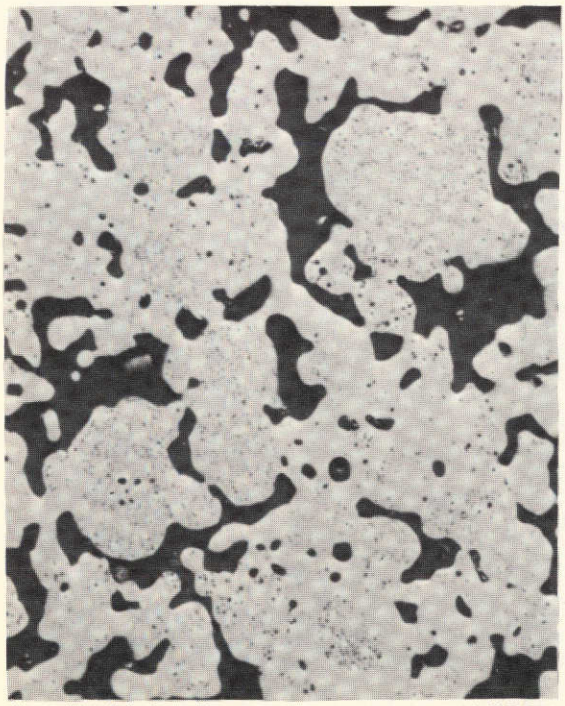
A. As-Impacted



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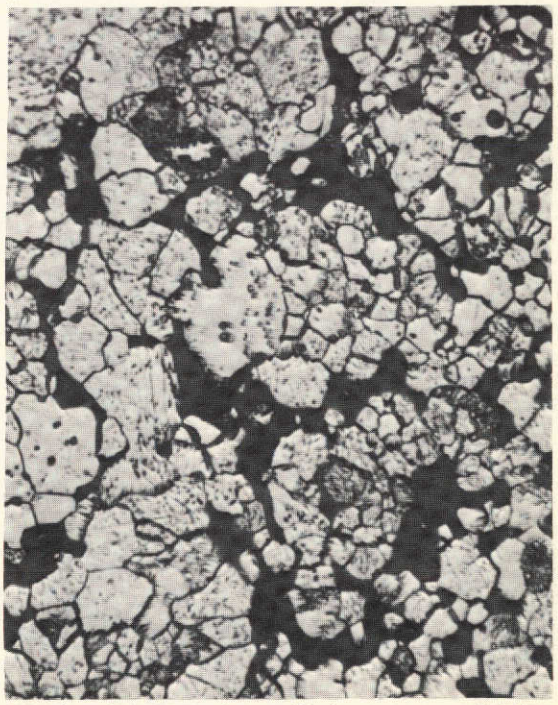
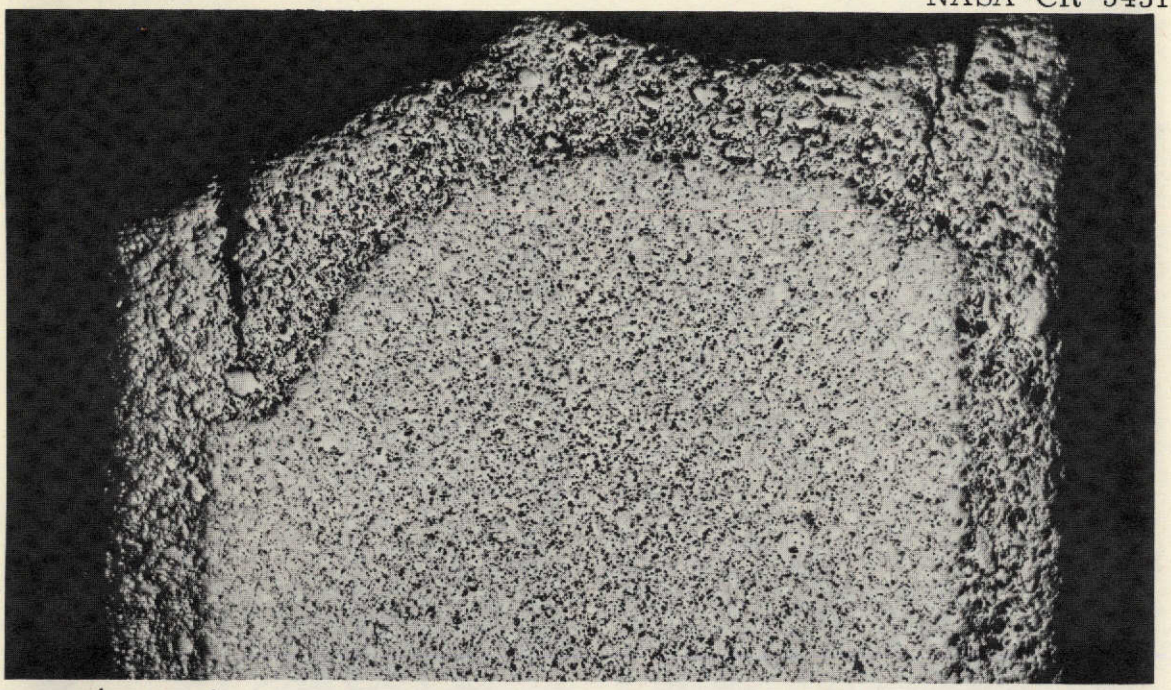


Etched (Murakami's) 1000X

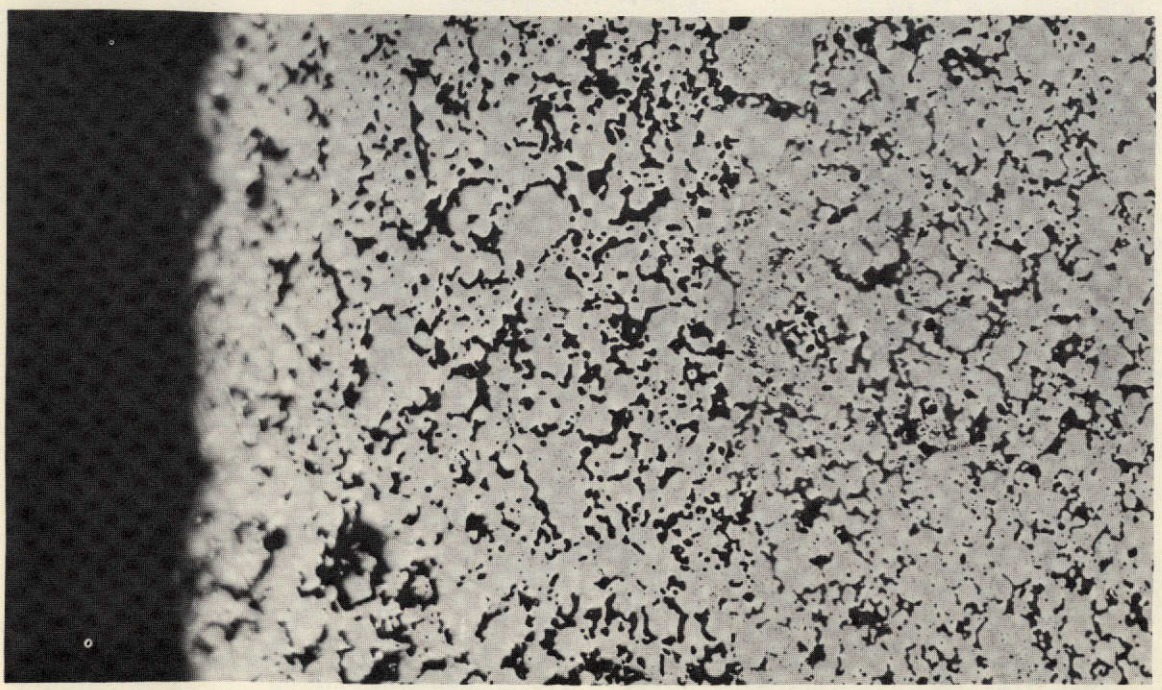
B. 2500 C for 1 hr in Vacuum

FIGURE 19

Impacted 20 vol% UO_2–W Plate Before and After 2500 C Anneal
Neg. 4Z-5109A; 4Z-5109B; 4Z-5109C; 4Z-5109D



~0.012 in. 50X



No UO_2 (~0.012 in.) UO_2 Still in Tact 250X

FIGURE 20

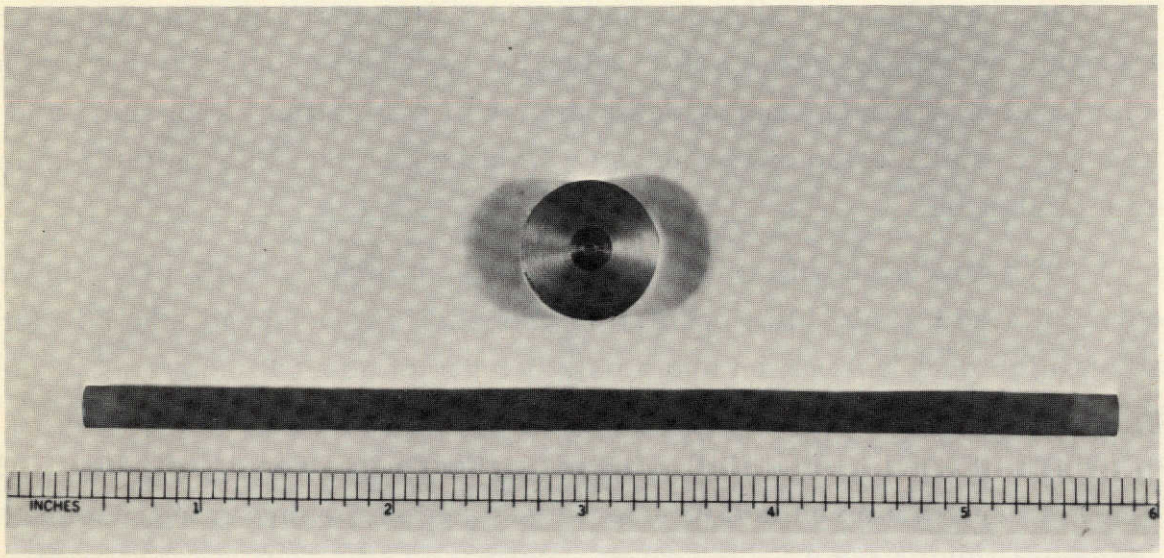
UO_2-Depleted Zone in 20 vol% UO_2–W Impacted Plate
After a 2500 C for 1 hr Vacuum Anneal
Neg. 5Z-5109A; 5Z-5109B

Fabrication by Extrusion*

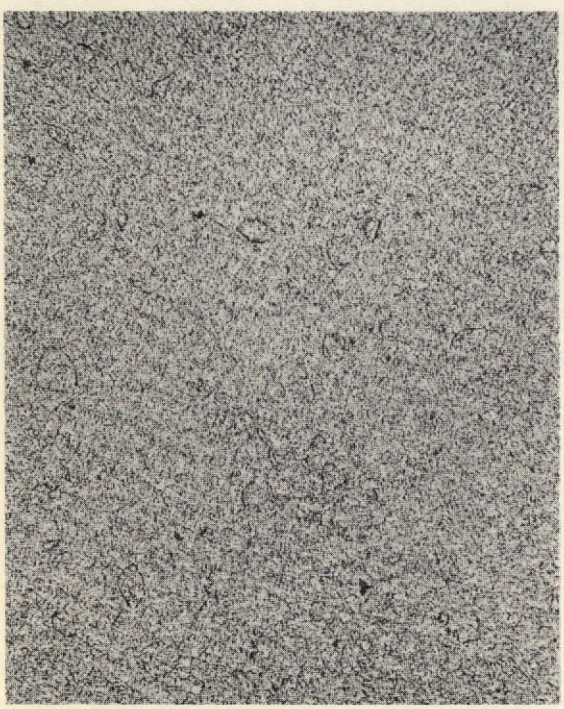
In previous work it had been demonstrated that a powder metal compact could be worked into a solid, metallic, wrought product by the extrusion process. Extrusion provides a means of evaluating stringering and the effect recrystallization of the tungsten would have upon UO_2 particle size and distribution.

The powder used in this experiment contained 20 vol% UO_2 and was coprecipitated by the acid flocculation process. As-reduced, the powder had an olive drab coloration and a bulk density of about 6.33 g/cm^3 (36% theoretical density). The powder was isostatically compacted at 80,000 psi to form a small billet having a theoretical density of about 75%. The billet was dry machined to size, given a final cleanup in ultra-pure hydrogen at 900 C for 3 hr, and vacuum sealed within a heavy wall molybdenum can. The thick molybedum can served the dual purpose of • reducing chilling during transfer from the preheat furnace to the press and • providing the required stiffness for good flow characteristic at the extrusion temperature. The billet was induction heated to 1875 C and extruded at a 9:1 ratio producing a dense, wrought product. Although the extrusion temperature used was above the normal sintering temperature, the billet was only allowed to soak at this temperature for 5 min, and the total heating time from room temperature to extrusion was only 25 min. Figure 21 shows a cross-section of the extruded rod and a short length of the cermet core. A hot acid stripping bath, containing equal parts of water, 70% HNO_3, and 93% H_2SO_4, was used to remove the molybdenum from the cermet core. Low magnification shows that the dispersion of the UO_2 is quite uniform; higher magnification (Figure 22) better shows the structure obtained.

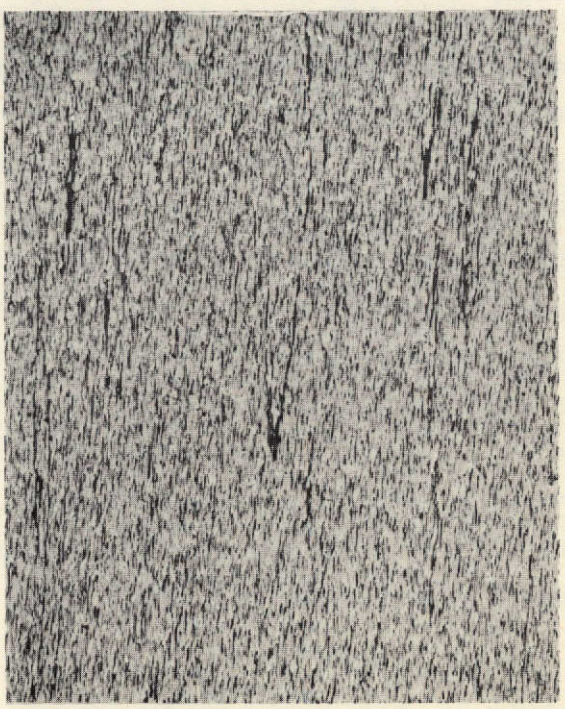
* Performed by G. S. Allison, Metallurgy Development, Battelle-Northwest.



A. As-Extruded: Extrusion Temperature 1875 °C, Extrusion Ratio 9:1



As-Polished 100X
B. Transverse Section

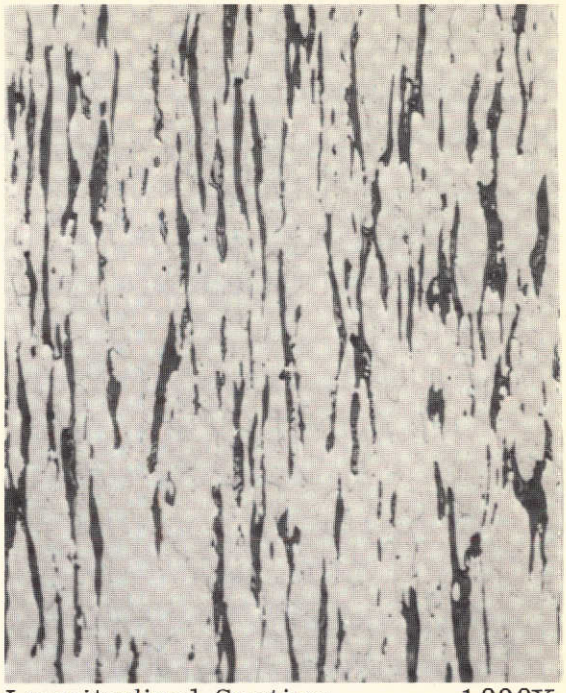


As-Polished 100X
C. Longitudinal Section

FIGURE 21

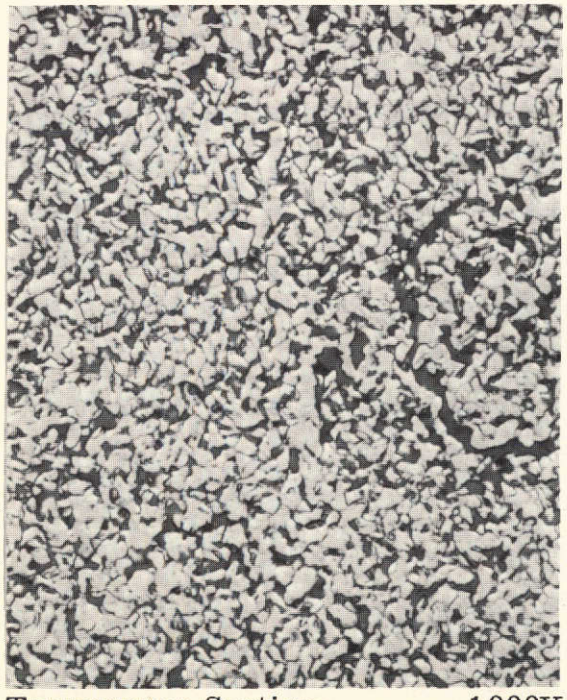
Rod Extrusion Having a 20 vol% UO_2–W Core
Neg. 5Z-5094; 4Z-4594A; 4Z-4592A

Transverse Section 1000X



Longitudinal Section 1000X

As-Polished



Transverse Section 1000X

Longitudinal Section 1000X

Etched (Murakami's)

FIGURE 22

20 vol% UO_2–W, As-Extruded

Neg. 4Z-4592B; 4Z-4594B; 4Z-4592D; 4Z-4594D

The following observations can be made from the photomicrographs.

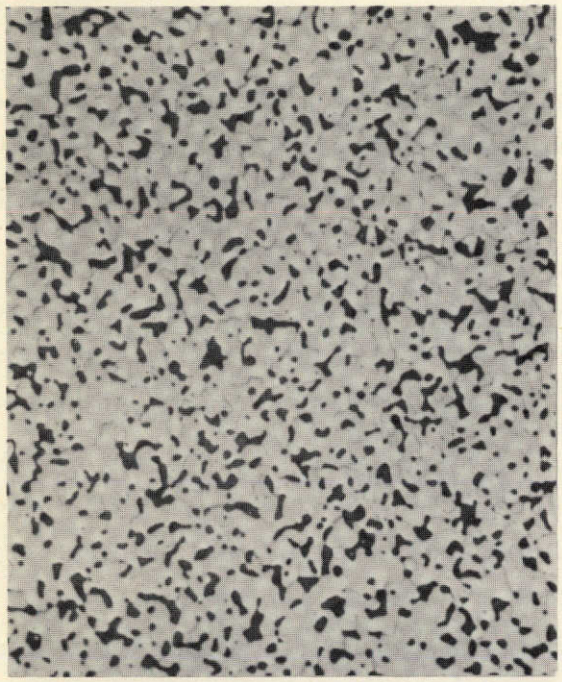
- No porosity in either the UO_2 or tungsten phase is evident indicating a near fully dense structure was obtained.
- Plastic deformation of the UO_2 occurred during extrusion.
- Nearly all of the UO_2 particles are 5 μ or less in diameter, 45 μ or less in length, and quite irregular in shape. The typical particle appears to be 2 to 3 μ in diameter by 20 to 30 μ in length with sharp feathered edges. The length to diameter ratio is less than the 27:1 ratio a pure plastic material would exhibit if extruded at 9:1 ratio. This leads one to conclude that at the extrusion temperature the UO_2 is stiffer than the tungsten and thus undergoes less deformation. Based upon the approximation that for pure plastic deformation the length over diameter ratio of the extruded particle is equal to the extrusion ratio raised to the 3/2 power, i.e. $L_E/D_E \approx R^{3/2}$, the extrusion ratio for the UO_2 phase was approximately 4.6:1. From this extrusion ratio for the UO_2 phase and the relationship $D_{Initial} \approx D_E\sqrt{R}$, the UO_2 just prior to extrusion should have had an average particle size of 6 to 7 μ. Although the initial particle size is unknown, compacts from powders produced by the same process normally have not had the UO_2 agglomerates larger than 2 to 4 μ. Therefore, considerable agglomeration of the UO_2 must have occurred during the relatively short time the billet was at the extrusion temperature.
- The lack of a complete network of sharp grain boundaries of the tungsten phase, as brought out in the etched condition, indicates a partially worked material that has not fully recrystallized. It should be noted; however, that the UO_2 stringers have restricted the growth of the new grains to primarily the axial direction thereby, interfering with the formation of a more equiaxed grain structure one would expect in the recrystallized areas.
- Typical grain size of the tungsten is 3 to 5 μ in diameter by 15 to 30 μ in length. The tungsten grains also are irregular in shape but definitely have a more equiaxed shape than that of the UO_2. The fact that

the typical length to diameter ratio is less than 27:1 ratio is further evidence that some recrystallization has occurred during or after the material passed through the die.

To evaluate the effect that high temperature treatment would have upon UO_2 size and distribution, small lengths of the as-extruded rod were annealed in vacuum. One sample was held at 2000 C for 1 hr and one was held at 2500 C for 1 hr. Figures 23 and 24 show that the high temperature anneal has had the following effects:

- Annealing has tended to partially spherodize the UO_2 causing some of the stringers to break up. The degree to which this has occurred increased with increasing temperature. UO_2 diameter, however, is still in the 2 to 3 μ range; whereas, the typical length has shortened to 10 to 30 μ.
- The tungsten phase appears to have fully recrystallized during the 2500 C anneal and nearly so in the 2000 C anneal.
- The tungsten grains have grown slightly yet still remain in the 4 to 5 μ in diameter range. High temperature treatment has reduced the typical length to the 10 to 15 μ range. The grains still remain irregular in shape.

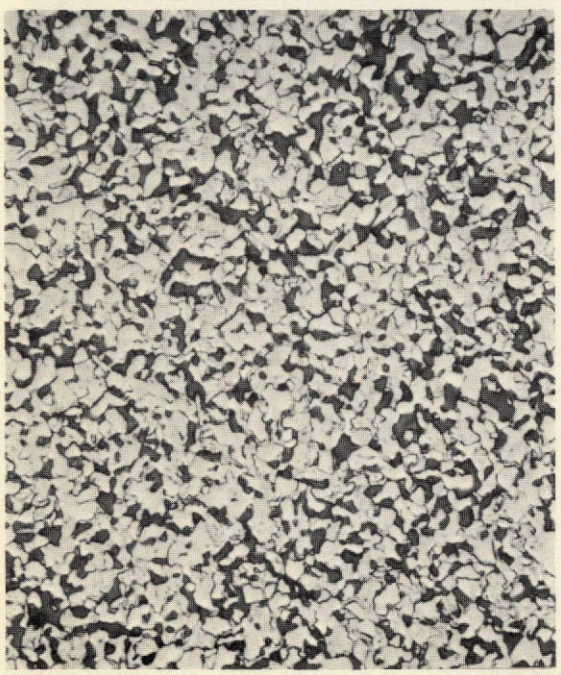
It is of interest to note that very little UO_2 was lost from the outer surface of the rod even though the molybdenum jacket had been removed chemically prior to annealing. Even in the more severe anneal, UO_2 loss was detected within only 0.001 in. of the surfaces. This is a considerable improvement compared to the loss encountered in the impacted plate under the same conditions. UO_2 losses from the exposed ends, however, were comparable to those of the impacted plate as can be seen by comparing Figures 20 and 25. At the higher magnification in Figure 25 one can find stringers, the base of which are still filled with UO_2, suggesting that the UO_2 is lost by vaporization from the surface of the exposed particle.



Transverse Section 1000X

Longitudinal Section 1000X

As-Polished



Transverse Section 1000X

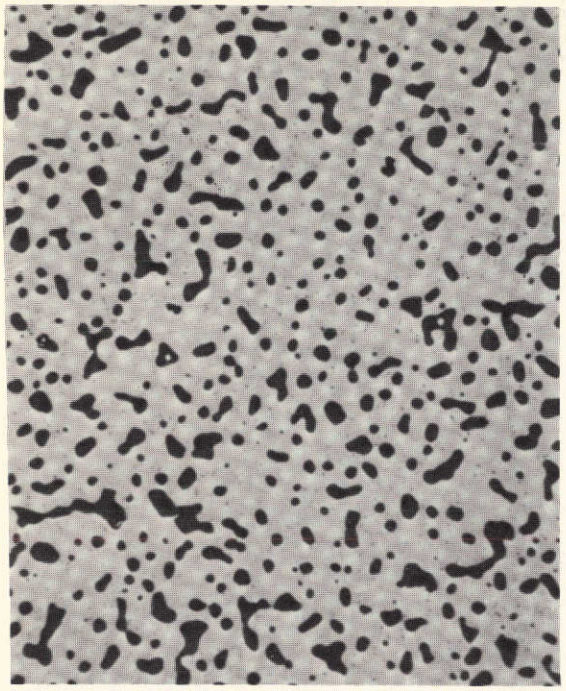
Longitudinal Section 1000X

Etched (Murakami's)

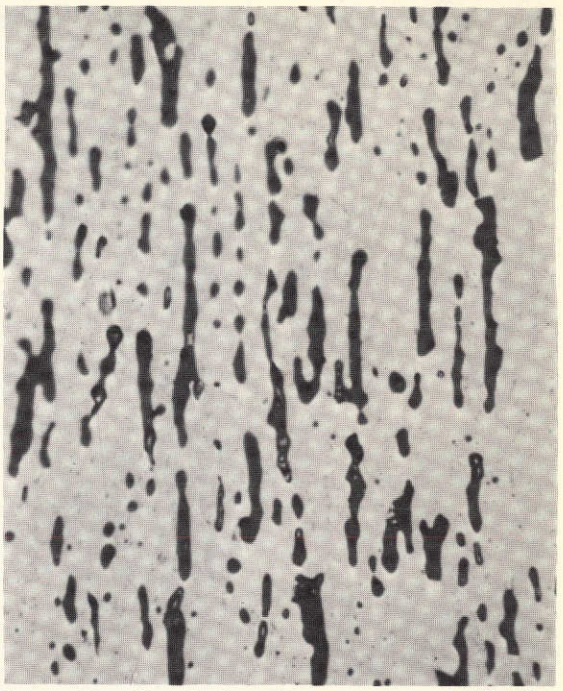
FIGURE 23

20 vol% UO_2-W Extruded Rod
(2000 C Vacuum Anneal for 1 hr)

Neg. 4Z-4894D; 4Z-4893E; 4Z-4894F; 4Z-4893G

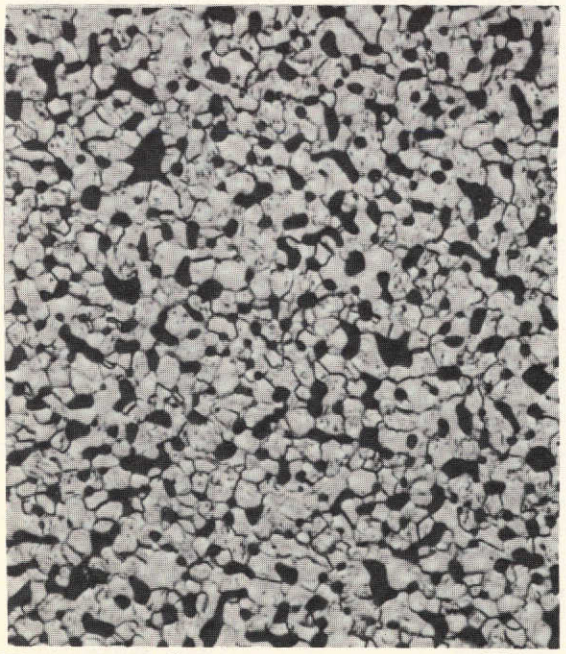


Transverse Section 1000X



Longitudinal Section 1000X

As-Polished



Transver Section 1000X

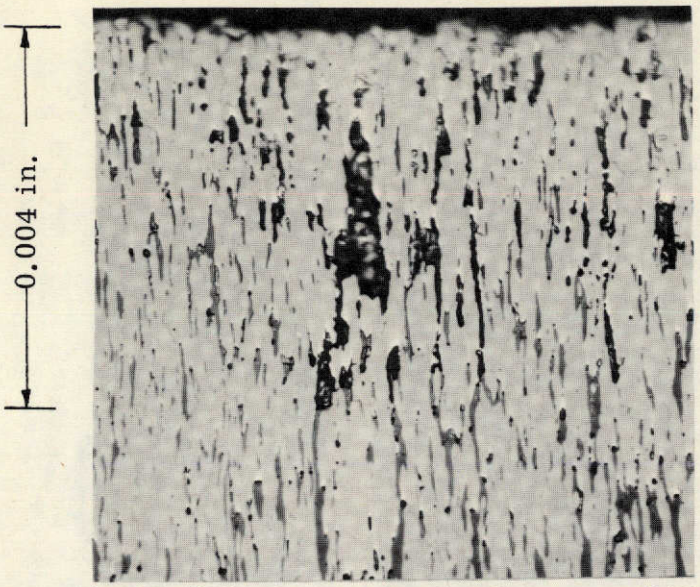
Longitudinal Section 1000X

Etched (Murakami's)

FIGURE 24

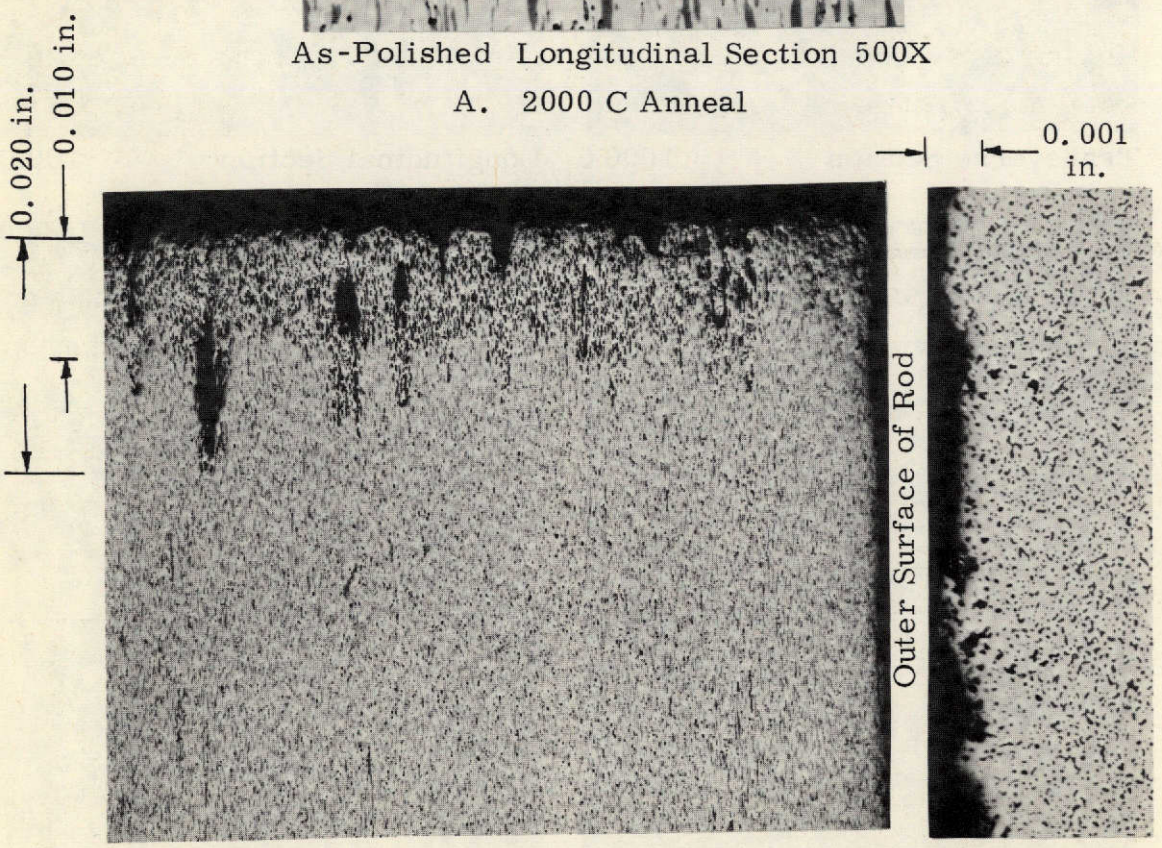
20 vol% UO_2-W Extruded Rod
(2500 C Vacuum Anneal for 1 hr)

Neg. 4Z-5105D; 4Z-5106C; 4Z-5105E; 4Z-5106D



As-Polished Longitudinal Section 500X

A. 2000 C Anneal



As-Polished Longitudinal Section 50X

As-Polished Transverse Section 250X

B. 2500 C Anneal

FIGURE 25

UO_2 Loss from Extruded Rod During 1 hr Vacuum Anneal

Neg. 4Z-4893C; 5Z-5106

DISCUSSION OF RESULTS

The primary objective of this program was to develop a process by which a submicron dispersion of UO_2 could be produced within a tungsten matrix. The feasibility of such a process was confirmed early in the program. In fact, such a dispersion was obtained in the first material made (Figure 2). This initial material, produced using the pH 9 process, was reduced readily to a metallic gray product at 900 C and was impacted at 1200 C to form a dense specimen for evaluation. Although the dispersed phase was submicron in size, a more uniform distribution of the UO_2 in the precipitates product was desired. This was accomplished by the development of the acid flocculation process in which a homogeneous precipitate was obtained. Although better distribution was obtained in the precipitation process, the product exhibited discoloration and pyrophoric tendencies in the reduction step. Considerable time was expended evaluating and trying to eliminate this apparent discoloration. The cause for the discoloration was not established; however, it is concluded that the problem is only a color phenomenon and not the result of impurities or incomplete reduction.

All of the material produced under this program exhibited thermal instability in that at elevated temperature the dispersed phase tended to agglomerate and coalesce to the point that the dispersion no longer was submicron. This phenomenon was observed in the treating of both loose powder and densified compacts. Material produced by the acid flocculation process, however, tended to be more sensitive to agglomeration in the loose powder state than the powder made by the basic process. The sensitivity of the powder to agglomerate not only varied from batch to batch but also varied in material within the same batch (Figure 11). Varying of the fuel loading within the 10 to 30 vol% UO_2 range did not affect the thermal stability of the product. It was found that treating the powder at 1300 to 1500 C tended to homogenize the structure of the product by causing the less sensitive material to agglomerate.

This sensitivity phenomenon is of paramount importance and must be given consideration when attempting to fabricate the powder into a useable metallic shape. Unless this sensitivity can be controlled or eliminated, one is restricted to fabrication techniques that employ either low temperatures or ones in which the time at elevated temperature is relatively short.

Both pneumatic impaction and fabrication by extrusion were successfully demonstrated with the coprecipitated powder. The gross agglomeration seen in the photomicrographs of the impacted plate was a property of the reduced powder and not attributible to the impaction process. The single attempt to fabricate this powder by extrusion demonstrated that a solid, metallic, wrought product could be produced. Annealing of the as-extruded material caused the UO_2 to spherodize partially, thereby minimizing the consequences of stringering. UO_2 loss from the extruded surface was very nominal. When the material was heated to 2500 C for 1 hr in vacuum, only the outer mil of material suffered any UO_2 loss. However, no attempt was made to compare this material with other materials under thermal cycling conditions.

RECOMMENDATIONS

Continuation of the program is recommended on the basis that the feasibility of producing UO_2-W powder by this process has been demonstrated and that the possible advantages the product has over powder blended material (as outlined in the Introduction) still are valid. In a continuation of the program special emphasis should be given to the following.

1. Improved Quality of the Reduced Product

   The product produced to date has exhibited variability in its sensitivity to agglomerate. Further development is needed to isolate the variables that led to this sensitivity. The process should be modified where possible to minimize product variability.

2. Evaluation of Additives

The addition of ThO_2, CaO, or Y_2O_3 to UO_2 is known to influence the manner in which the excess uranium formed at high temperatures is precipitated in UO_2. The addition of thorium, calcium, or yttrium during coprecipitation should be investigated and the resultant product evaluated in terms of thermal stability of the dispersion and fuel loss on thermal cycling.

3. Fabrication of Test Specimens

(a) The initial investigation of fabrication by extrusion was very encouraging. Further investigation is needed to evaluate the effects working and recrystallization have on the structure and fuel loss characteristics of the material. A program to establish rolling techniques starting with extruded material should be conducted.

(b) Preliminary data indicate that a small UO_2 particle size as well as additives is effective in reducing UO_2 loss during thermal cycling. Plates fabricated from coprecipitated material by various processes should be evaluated in terms of UO_2 loss characteristics during thermal cycling.

(c) Elevated temperature mechanical properties should be determined on fabricated material having a desired microstructure and density.

APPENDIX

X-RAY FLUORESCENCE ANALYSIS OF URANIUM AND TUNGSTEN IN THE SAME SOLUTION

W. G. Jolley*

Uranium and tungsten are high atomic number elements and their analysis by X-ray fluorescence requires standard equipment.

A procedure was developed for analysis of uranium and tungsten in the same solution. The two lines chosen for detection of these elements were U-$L_{\alpha 1}$ at 26.14-2θ and W-$L_{\alpha 1}$ at 43.02-2θ. These lines were well separated and the small amount of higher order interference was effectively lessened by pulse height discrimination. Scintillation counting is very efficient for these two elements and was used for the procedure. A working curve was made for each element by plotting a peak to background ratio (I_w/I Bkg or I_u/I Bkg) versus known standard values. The method is suitable for tungsten in the range of 0 to 16 g/liter and for uranium in a range from 0 to several percent. There would be no problem in altering the procedure for much higher concentrations of either element.

The detection limits were 0.03 g/liter for uranium and 0.07 g/liter for tungsten. The uranium analysis is accurate to ± 0.03 g/liter and the tungsten to ± 0.2 g/liter.

A standard General Electric XRD-5 X-ray unit equipped with fluorescence attachments and a linear amplifier and pulse height selector was the equipment used. This method of analysis used a lithium fluoride analyzing crystal, a chromium target X-ray tube, and scintillation counter. The X-rays were generated by 50 kV peak and 40 mA. The liquid samples were enclosed in 1/2 in. diam cells covered by 1/2 mil Mylar.

---

* Analytical Laboratory, Production Fuels Section, Irradiation Processing Department, General Electric Company, Richland, Washington.

ANALYSES OF STARTING MATERIALS

Ammonium Paratungstate	Lot 549B
WO_3	89.20%
Loss on Ignition	10.80%

Impurity Content, ppm

Aluminum	0.5	Molybdenum	11
Calcium	2	Nickel	<1
Chromium	<1	Nonvolatile Matter	<50
Copper	<0.1	Silicon	<1
Iron	<1	Sodium	14
Manganese	<2		

Uranium — Dingot 223B

The uranyl nitrate solution was prepared by the dissolution of metallic uranium in CP HNO_3.

Impurity Content, ppm:

Aluminum	20	Lithium	<1
Bismuth	<0.5	Magnesium	14
Boron	<0.1	Manganese	<10
Cadmium	<0.2	Molybdenum	<5
Carbon	20-30	Nickel	17
Chromium	6	Phosphorus	<50
Cobalt	<5	Silicon	11
Copper	3	Sodium	<10
H_2	2.1	Tin	<5
Iron	45	Vanadium	<20
Lead	<5	Zinc	<20

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HANFORD ATOMIC PRODUCTS OPERATION
SPECTROCHEMICAL LABORATORY – BUILDING 3706
SPECTROCHEMICAL ANALYSIS REPORT

DATE REPORTED:

SPEC. LAB. NO. SP	SUBMITTER'S NO.	ANALYZED BY
MATERIAL: Tungsten - UO_2	PLATE NO.	SUBMITTED BY

REMARKS: Typical analysis of as-reduced powder prepared by coprecipitation.

ELEMENT		ELEMENT		ELEMENT		ELEMENT		ELEMENT	
A		Cu	T	Le	–	Pt		Th	
Ag	–	Dy		Li	T	Ra		Ti	–
Al	T	Er		Lu		Rb		Tl	
Az	–	Eu		Mg	T	Re		Ta	
AuA	–	Fe	T	Mn	T	Rh		U	S
B	–	Ga		Mo	–	Rn		V	–
Ba	–	Gd		N		Ru		W	S
Be	–	Ge		Na	–	Sb	–	Xe	
Bi	–	H		Nd		Sc		Y	
C	*	He		Ne		Se		Yb	
Ca	–	Hf		Ni	–	Si	T	Zn	–
Cb	–	Hg	–	O		Sm		Zr	
Cd	–	Ho		Os		Sn	T		
Ce		In		P	–	Sr	–		
Co	T	Ir		Pb	–	Ta	–		
Cr	–	K	–	Pd		Tb			
Cs		Kr		Pr		Te			

SYMBOL	MEANING	APP'X. CONC.
S	STRONG	GREATER THAN 1 %
M	MODERATE	1% TO 0.01%
T	TRACE	LESS THAN 0.01%
+	DETECTED	MINOR CONSTITUENT

SYMBOL	MEANING
L	LESS THAN
G	GREATER THAN
	ELEMENT NOT INVESTIGATED
–	ELEMENT NOT DETECTED
*	INTERFERENCE
T *	DETECTION UNCERTAIN, INTERFERENCE

SYMBOL	MEANING
NUMERICAL VALUES	PARTS PER MILLION
L100 *	EXAMPLE IN WHICH LOWER LIMIT OF 100 PPM. IS SET BY REAGENT OR SPECTRUM INTERFERENCE
L100 –	EXAMPLE IN WHICH 100 PPM. IS LIMIT OF SENSITIVITY

REMARKS:

REPORT APPROVED –

A-4361-138 (9-58)

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